

Results of LLNL's Participation in the 16th OPCW Proficiency Test

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January 27, 2005

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This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

Results of LLNL's Participation in the 16th OPCW Proficiency Test

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In the summer and fall of 2004, LLNL scientists prepared samples for the 16th official Organisation for the Prohibition of Chemical Weapons (OPCW) Proficiency Test.

Our report of this effort is attached.

This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.



ORGANISATION FOR THE PROHIBITION OF CHEMICAL WEAPONS

Report of the Sixteenth Official OPCW Proficiency Test

Part I: Sample Preparation

Laboratory code: Sample Prep Lab

Total number of pages: 128

¹ Total number of pages including cover page and all attachments

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1 Introduction

The Sixteenth Official OPCW Proficiency Test started in October 2004. The samples were prepared by scientists affiliated with the Forensic Science Center at the Lawrence Livermore National Laboratory in Livermore, California, USA. The work was funded by the US Department of Energy.

The test scenario and the spiking and background chemicals were discussed and agreed in advance with the OPCW. The samples were prepared in accordance with "Work Instruction for the Preparation of Test Samples for OPCW Proficiency Tests" (Document No.: QDOC/LAB/WI/PT2).

The preparation of the test samples and their analysis are described in this report.

2 Test Scenario

A State Party has presented in accordance with Article IX of the Convention an inspection request for a challenge inspection. The Executive Council did not decide against this request, and the Director-General, in accordance with Part X of the Verification Annex, sent a challenge inspection team to a certain facility that has been accused of producing chemical warfare agents.

The facility is described by the inspection team as being a chemical research plant with well-equipped analytical and synthesis laboratories. The inspection team collected one sample inside the analytical laboratory from a container labeled "Organic Waste". Another sample was collected outside a synthesis laboratory from a large unlabeled plastic container, "Liquid". The inspection team collected a third sample, "Soil", outside the facility's chemical loading/unloading area. The inspection team did not analyze the samples on-site and forwarded them directly for off-site analysis. In addition, the inspection team could not collect corresponding blanks and used pure organic solvents and pre-cleaned soil similar to that found at the facility.

Please analyze the samples for the presence of any Scheduled chemicals and/or their degradation/reaction products, taking into account the characteristics of the samples.

3 List of Scheduled Chemicals

The majority of the chemicals used in this Proficiency Test were obtained commercially. Several spiked chemicals (both scheduled and unscheduled) were synthesized in-house. The purities of the scheduled chemicals were determined by NMR (¹H and ¹³C{¹H}) and those results are presented in Part II, *Purity Checks*, of this report.

The Scheduled chemicals spiked into the three matricies are presented in the table on the next page.

List of Spiking Chemicals for the Sixteenth Official OPCW Proficiency Test

Sample code	No	Compound name	CAS number	Spiking Chemical Structure	Schedule number
O	A	Trichloronitromethane	76-06-2	CI NO ₂	3.A.04
0	В	2-(N-Ethyl-N-propylamino)ethylchloride		CI	2.B.10
О	C	2-(N,N-Diisopropylamino)ethylchloride	96-79-7	CI	2.B.10
0	D	2-(N,N-Diethylamino)ethylchloride	100-35-6	CI	2.B.10
L	E	Triethanolamine	102-71-6	ОН	3.B.17
L	G	2-(N,N-Diisopropylamino)ethanol	96-80-0	но	2.B.11
S	Н	Ethyldiethanolamine	139-87-7	ОН	3.B.15
S	I	Methyldiethanolamine	105-59-9	ОН	3.B.16
S	J	Triethanolamine	102-71-6	он N ОН	3.B.17

4 Sample Preparation

Several preliminary stability studies were carried out in the months preceding the 16th Official Proficiency Test. These studies were used to down-select the list of scheduled chemicals used for spiking and helped to determine stable amounts of materials and containers. Based on these studies, and in consultation with OPCW, three sample matricies were spiked as shown below.

4.1 Organic sample (O)

The following chemicals constituted the Organic sample.

Component	Origin/purity	Concentration (mg/L)
Trichloronitromethane	In-house synthesis	30
2-(N-Ethyl-N-propylamino)ethylchloride	In-house synthesis ¹	10
2-(N,N-Diisopropylamino)ethylchloride	Aldrich ¹ , 97%	6
2-(N,N-Diethylamino)ethylchloride	Aldrich ¹ , 99%	30
Diesel fuel	Local gas station ¹	200
Carbon tetrachloride	J.T. Baker, 100%	20
2,4-Dinitro-t-butylbenzene	In-house synthesis, 97%	40
2,4-Dinitrotoluene	Eastman Kodak	20
2-Nitroaniline	Eastman Kodak	20
Hexane	Fluka, min. 99.5%	Solvent

¹The preparation of the chlorides and diesel fuel cleanup are presented in section 4.4.

4.2 Liquid sample (L)

The following chemicals constituted the Liquid sample.

Component	Origin/purity	Concentration (mg/L)
Triethanolamine	Aldrich, 98%	500
2-(N,N-Diisopropylamino)ethanol	Aldrich, 99%	30
2-(N,N-Diethylamino)ethanol	Aldrich, 98%	20
2-(N,N-Dimethylamino)ethanol	Aldrich, 99%	20
2-(N-Butyl-N-methylamino)ethanol	In house synthesis	30
2,6-Dinitroaniline	Aldrich, 97%	20
2-Nitroaniline	Eastman Kodak	20
Pyridine	Sigma Aldrich, 99.9%	20
Poly(ethyleneglycol), M _n 200	Aldrich	200
Methanol	Riedel-deHaën,	Solvent
	Chromasolv®, min 99.9%	

The following chemicals constituted the Soil sample.

Component	Origin/purity	Concentration (mg/kg)
Ethyldiethanolamine	Aldrich, 98%	20
Methyldiethanolamine	Aldrich, 99.9+%	20
Triethanolamine	Aldrich, 98%	20
Diesel fuel	Local gas station ¹	200
Caffeine	Aldrich, 99%	40
Nicotine	Sigma, 99%	40
Malation	Chem Service, 99.2%	40
Tributylamine	EM, 98%	40
Tripentylamine	EM, 98%	40
1,3-Dinitrobenzene	Aldrich, 97%	40
2,4-Dinitrotoluene	Eastman Kodak	20
Dichloromethane	Aldrich, 99.9%	(dried)
Sand, purified	J.T. Baker	Matrix

¹The preparation of the chlorides and diesel fuel cleanup are presented in section 4.4.

4.4 Chemical preparation

The three scheduled chlorine-containing amines were purchased or synthesized as their hydrochloride salts. These salts were converted to the chloride form by adding 1.0 mL of aqueous sodium hydrogen carbonate to a known amount of material and extracting the formed chloride into three serial aliquots of 1.0 mL of hexane. The resulting hexane extracts were dried using a column of anhydrous sodium sulfate. To estimate the resulting solution concentrations, it was assumed that 100% conversions and recoveries of the chlorides into the hexane were accomplished.

Diesel fuel was obtained from a local gas station and washed prior to use. To wash the fuel, 100 mL of fuel was placed in a 500 mL round-bottomed flask and 100 mL of concentrated sulfuric acid was added. The mixture was stirred vigorously for 2 hours. The two phases were placed in a separatory funnel and the sulfuric acid layer was discarded. The fuel layer was washed sequentially with 20 mL water, 10 mL of a 5% solution of sodium hydrogen carbonate, and 20 mL of water. The washed fuel was dried over anhydrous sodium sulfate and finally dried over anhydrous sodium carbonate, yielding a clear, colorless liquid.

The remaining chemicals were obtained or synthesized neat and ready to use.

4.5 Protocols for preparing test samples

The soil samples and blank soil samples were prepared on October 6, 2004 and the two liquid samples and their corresponding blanks were prepared on October 7, 2004. Two staff members of the OPCW laboratory witnessed the sample preparation process. A total of 40 sets of samples and their corresponding blanks were made. Seventeen sets of samples and their corresponding blanks

were sent to laboratories that were planning to participate in the 16th OPCW Proficiency Test, two sets of samples and blanks were sent to the evaluation laboratory, and two sets of samples and blanks were sent to the OPCW laboratory. Three sets of samples and blanks, chosen randomly by the OPCW staff members, were used by the preparation laboratory for stability testing. An additional set was used by the preparation laboratory for qualitative analysis. The remaining sample sets were retained by the sample preparation laboratory for contingencies.

4.6 Organic sample (Code O) and Organic sample blank (Code OB) preparation

Hexane was added to a 500 mL, glass, solvent-rinsed, volumetric flask. Next, 125 microliters of diesel fuel was added to the hexane. Stock solutions of individual test chemicals were made in hexane and ranged in concentration from 1-30 mg/mL. A sufficient volume of each stock solution was added to the hexane solution to yield the desired analyte concentrations, listed in the previous section. The hexane solution was brought to a final volume of 500 mL and analyzed by GC/MS to verify that the solution was of the expected composition.

Neat hexane was used as the organic solvent blank. The hexane was analyzed by GC/MS to verify that it contained none of the added test chemicals.

4.7 Liquid sample (Code L) and Liquid sample blank (Code LB) preparation

Methanol was added to a glass, solvent-rinsed, 2.5 L jug. Next, 250 microliters of poly(ethyleneglycol) was added to the methanol. Stock solutions of individual test chemicals were made in methanol and ranged in concentration from 5-50 mg/mL. A sufficient volume of each stock solution was added to the methanol solution to yield the desired analyte concentrations, listed in the previous section. The methanol solution was brought to a final volume of 1.0 L and analyzed by GC/MS to verify that the solution was of the expected composition.

Neat methanol was used as the organic solvent blank. The methanol was analyzed by GC/MS to verify that it contained none of the added test chemicals.

4.8 Soil sample (Code S) and Soil sample blank (Code SB) preparation

To make the soil samples, the test chemicals were first added to dichloromethane. The resulting dichloromethane solution was added to the sand and allowed to evaporate, leaving the test chemicals on the soil.

Dichloromethane was added into a glass, solvent-rinsed, 2.5 L jug. Next, 625 microliters of diesel fuel was added to the dichloromethane. Stock solutions of individual test chemicals were made in dichloromethane and ranged in concentration from 10-20 mg/mL. A sufficient volume of each stock solution was added to the dichloromethane solution to yield the desired analyte concentrations, listed in the previous section. The dichloromethane solution was brought to a final volume of 1.25 L and analyzed by GC/MS to verify that the solution was of the expected composition.

Into each of five, solvent-rinsed, 1-L beakers were placed 500 g sand. Into each beaker containing sand, 250 mL of the above dichloromethane solution were added. The resulting mixture was stirred with a metal spatula and placed in a chemical fume hood. The dichloromethane was allowed to evaporate overnight, leaving the test chemicals on the sand. When the sand was dry, it was again

mixed in the beaker and transferred into an empty bottle, which had previously contained clean sand. The sand contained in all of the beakers (2500 g total) was transferred to the bottle. The bottle containing spiked sand was then placed on a roller and homogenized for approximately 5 minutes. The resulting spiked sand was then sampled, extracted, derivitized and analyzed by GC/MS to verify that it was of the expected composition.

Blank samples were prepared by exposing 2500g of sand to 1.25 L of dichloromethane. The blank sand was prepared in a manner identical to that of the spiked soil, with the exception that no test chemicals were added. The resulting blank sand was sampled, extracted, derivitized and analyzed by GC/MS to verify that it was free of test chemicals.

4.9 Standards

Two sets of standards were prepared, one each for the OPCW laboratory and the evaluation laboratory. These standards were prepared by dilution from the stock solutions in the range of 1 to 2 mg/mL. A set of vials of each standard, along with a description, was shipped a few days after the samples were shipped.

5 Packaging and Transportation

The sample preparation laboratory received the list of participating laboratories on 28 September 2004 and contacted them on several occasions to confirm delivery addresses and preferred shippers. It was gratifying that most laboratories responded promptly, however there were significant communication issues with several laboratories.

5.1 Announcement of Sample Dispatch

Annex A contains a copy of the fax and/or e-mail detailing the sample dispatch information.

5.2 Packaging

10 mL of the organic solution (hexane) and corresponding blank were pipetted into 20-mL vials (pre-cleaned borosilicate glass) and capped (Teflon-lined, silicon-backed seal). The caps were tightened, taped with a thin strip of duct tape followed by the application of a tamper indicating seal. These bottles were labeled and placed inside of plastic bottles with absorbent material. These secondary bottles were sealed with a heat-shrink plastic seal.

18 mL of the liquid solution (methanol) and corresponding blank were pipetted into 20-mL vials (pre-cleaned borosilicate glass) and capped (Teflon-lined, silicon-backed seal). The caps were tightened, taped with a thin strip of duct tape followed by the application of a tamper indicating seal. These bottles were labeled and placed inside of plastic bottles with absorbent material. These secondary bottles were sealed with a heat-shrink plastic seal.

50 grams of the soil and the soil blank were weighed into 2-oz. clear glass jar and sealed with caps (Teflon-bonded to polypropylene foam). The caps were tightened, taped with a thin strip of duct tape followed by the application of a tamper indicating seal. These bottles were labeled and sealed inside of a heat-sealable plastic bag.

All six containers (O, OB, L, LB, S, SB) were placed inside of a foam-lined cardboard box. Each box was accompanied by a letter (Annex B). The shipping information was affixed to the exterior of the box.

5.3 Confirmation of Dispatch

The LLNL shipping department took custody of the samples and provided tracking numbers. Participating laboratories were notified of the carrier and tracking number (see Annex C for an example). Due to multiple transportation restrictions, multiple carriers had to be used.

5.4 Sample Receipt

At least 10 of the 17 laboratories received the samples within one week of shipping. A summary of the shipping and receipt information is provided in Annex D.

6 Analysis of Samples

The three sample matrices were analyzed both quantitatively and qualitatively.

6.1 Quantitative Analysis

Each of the three matricies required different sample work up procedures.

6.1.1 Analytes in Organic Solution

Analysis of spiked chemicals in the organic solution was performed by direct injection of one microliter of sample solution into the GC/MS (EI). Quantitation was performed using extracted ion chromatograms and using heptadecane (present as a component of the diesel fuel background) as an internal standard. A three point calibration curve (10 to 40 mg/L) was prepared fresh for each sampling day.

m/z EICs used for quanitation

Cmpd #	Compound name	EIC m/z
A	Trichloronitromethane	117
В	2-(N-Ethyl-N-propylamino)ethylchloride	120
С	2-(N,N-Diisopropylamino)ethylchloride	106
D	2-(N,N-Diethylamino)ethylchloride	86

6.1.2 Analytes in Liquid Solution

Analysis of spiked chemicals in the organic solution was performed by direct injection of one microliter of sample solution (compound E was diluted with methanol before injection) into the GC/MS (EI). Quantitation was performed using extracted ion chromatograms and using PEG200 (present as a component added to the background) as an internal standard. A three point calibration

curve (for compound E: 50 to 200 mg/L; for compound G: 10 to 40 mg/L) was prepared fresh for each sampling day.

m/z EICs used for quantitation

Cmpd #	Compound name	EIC m/z
Е	Triethanolamine	118
G	2-(N,N-Diisopropylamino)ethanol amine	114

6.1.3 Analytes in Soil Sample

Analysis of spiked chemicals in the soil sample was performed by extraction, derivatization, and direct injection of one microliter of sample solution into the GC/MS (EI). Quantitation was performed using extracted ion chromatograms using heptadecane (present as a component of the diesel fuel background) as an internal standard. A three point calibration curve (10 to 40 mg/L) was prepared fresh for each sampling day and was derivatized in the same manner and at the same time as the sample extracts.

The extraction of the sand was accomplished by sonicating 2.0 g of sand with 2.0 mL of methanol for 15 minutes. The resulting solution was centrifuged for 3-5 minutes and 500 μ L of the clarified extract were transferred to a 4-mL vial. The extract was evaporated to dryness with a gentle stream of nitrogen and reconstituted immediately with 200 μ L of N, O-Bis(trimethylsilyl)trifluoroacetamide (BSTFA). The resulting solution was sealed and heated at 60°C for 30 minutes to allow derivatization of the three amines.

m/z EICs used for quanitation

_							
	Cmpd #	Cmpd # Compound name					
	Н	Ethyldiethanolamine	174				
ĺ	Ι	Methyldiethanolamine	160				
Ī	J	Triethanolamine	262				

6.1.4 Quantitative Analysis Results

Chemical A: Trichloronitromethane (mg/L)

	CIIC	1111041111	I I I CHIOI			, 2)	
Sample	Day 0	Day 7	Day 11	Day 15	Day 21	Day 29	Day 40
O/25	30	25	26	32	26	26	26
O/35	33	30	24	30	26	25	27
O/40	33	28	29	30	26	26	31
Average	32	28	26	31	26	26	28
%RSD	5.4%	9.1%	9.6%	3.8%	1.0%	0.9%	8.0%

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Chemical B: 2-(N-Ethyl-N-propylamino)ethylchloride (mg/L)

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Sample	Day 0	Day 7	Day 11	Day 15	Day 21	Day 29	Day 40
O/25	10.4	10.6	10.7	11.0	10.4	10.7	10.2
O/35	10.9	11.5	10.2	10.4	10.9	9.5	10.7
O/40	11.3	11.6	11.4	10.7	11.4	10.1	11.4
Average	10.9	11.2	10.8	10.7	10.9	10.1	10.8
%RSD	4.2%	4.7%	5.4%	2.9%	4.5%	6.0%	5.7%

Chemical C: 2-(N,N-Diisopropylamino)ethylchloride (mg/L)

Sample	Day 0	Day 7	Day 11	Day 15	Day 21	Day 29	Day 40
O/25	5.3	5.2	5.4	5.2	4.8	5.8	5.2
O/35	6.0	5.6	5.2	5.0	5.1	5.1	5.4
O/40	6.5	5.7	5.8	5.2	5.2	5.5	5.7
Average	5.9	5.5	5.5	5.1	5.1	5.5	5.4
%RSD	9.9%	4.2%	5.3%	2.1%	3.9%	5.8%	4.8%

Chemical D: 2-(N,N-Diethylamino)ethylchloride (mg/L)

Chemical D. 2 (1311 Diethylamino)ethylemoriae (mg/L)							
Sample	Day 0	Day 7	Day 11	Day 15	Day 21	Day 29	Day 40
O/25	24	23	23	25	25	23	24
O/35	25	25	22	24	26	21	25
O/40	25	25	24	24	27	22	26
Average	25	24	23	24	26	22	25
%RSD	3.8%	5.9%	4.9%	3.8%	4.6%	4.7%	4.6%

Chemical E: Triethanolamine (mg/L)

Sample	Day 0	Day 7	Day 11	Day 15	Day 21	Day 29	Day 40
O/25	433	374	384	418	426	516	500
O/35	487	490	477	409	476	564	557
O/40	530	516	461	482	506	512	579
Average	483	460	441	436	469	531	545
%RSD	10%	16%	11%	9.0%	8.6%	5.5%	7.5%

Chemical G: 2-(N,N-Diisopropylamino)ethanol (mg/L)

Chemical G. 2 (1911 Busopi opylamino) chanoi (mg/L)								
Sample	Day 0 Day 7 Day 11		Day 15	Day 21	Day 29	Day 40		
O/25	30	28	29	33	33	28	26	
O/35	32	29	25	32	31	26	30	
O/40	33	30	27	31	31	28	29	
Average	32	29	27	32	32	28	28	
%RSD	4.6%	3.2%	7.6%	4.0%	4.3%	4.5%	7.7%	

Chemical H: Ethyldiethanolamine (mg/kg)

Sample	Day 0	Day 8	Day 12	Day 16	Day 22	Day 30	Day 40
O/25	10.3	8.6	7.5	10.9	7.9	10.0	9.3
O/35	11.6	9.6	10.9	9.7	8.1	10.0	10.2
O/40	10.2	10.3	10.8	11.8	11.8 7.3		10.8
Average	10.7	9.5	9.7	10.8	7.8	10.2	10.1
%RSD	7.2%	8.8%	20%	9.7%	5.2%	2.3%	7.6%

Chemical I: Methyldiethanolamine (mg/kg)

chemical it witch year chanolamine (mg/kg)									
Sample	Day 0	Day 8	Day 12	Day 16	Day 22	Day 30	Day 40		
O/25	7.5	7.6	7.4	6.6	5.3	7.0	7.3		
O/35	9.3	7.9	8.2	6.2	5.5	6.8	7.7		
O/40	8.3	7.7	7.7	7.8	4.5	7.2	7.7		
Average	8.4	7.7	7.8	6.9	5.1	7.0	7.6		
%RSD	11%	2.1%	5.4%	12%	10%	3.1%	3.0%		

Chemical J: Triethanolamine (mg/kg)

Sample	Day 0	Day 8	Day 12	Day 16	Day 22	Day 30	Day 40
O/25	8.7	10.4	9.2	11.3	8.1	12.5	12.7
O/35	9.6	10.9	9.5	8.7	8.0	12.3	13.0
O/40	6.6	13.0	8.9	12.1	6.9	12.9	12.8
Average	8.3	11.4	9.2	10.7	7.7	12.6	12.8
%RSD	19%	12%	3.3%	17%	8.2%	2.2%	1.1%

OPCW 6.2 Qualitative Analysis

Sample Prep, Part I, Page no. <u>14</u>

The results of the qualitative analysis are presented in Part III, *Qualitative Analysis*, of this report. That section of the report is documented using the standard forms for reporting results of an Official OPCW Proficiency Test.

The Organic samples were analyzed directly by GC/MS EI & CI. The Liquid samples were analyzed after derivitization with BSTFA to form the trimethylsilyl derivative and then analyzed by GC/MS EI & CI. The Soil samples were first extracted with methanol, derivitized with BSTFA to form the trimethylsilyl derivative and then analyzed by GC/MS EI & CI.

Detailed procedures are described in Part III of this report.

Annex A: Dispatch Announcement



October 6, 2004

University of California Lawrence Livermore National Laboratory 7000 East Ave Livermore, CA 94550

Date of Sample Dispatch for the Official 16th OPCW Proficiency Test

Dear Colleagues,

The samples for the 16th Official OPCW Proficiency Test will be dispatched from our laboratory on **8 October 2004**. FedEx or other air fright carrier will deliver the packages. The airway bill numbers will be released on the day of sample dispatch. The test samples contain an organic sample, liquid sample, and soil with their corresponding blanks.

Please acknowledge the receipt of the fax or email to:

Fax: (925) 423-9014 Email: alcaraz1@llnl.gov

Good luck with the analysis.

Armando Alcaraz

Fax: (925) 423-9014

Sincerely, Armando Alcaraz Forensic Science Center Lawrence Livermore National Laboratory Voice: (925) 423-6889

Annex B: Letter Accompanying Package



October 8, 2004

University of California Lawrence Livermore National Laboratory 7000 East Ave Livermore, CA 94550

Participating Laboratories 16th Official OPCW Proficiency Test

TEST SAMPLES FOR THE 16TH OFFICIAL OPCW PROFICIENCY TEST

Dear Colleagues,

- Please find enclosed samples for chemical analysis according to the information which you received from the OPCW regarding the 16th OPCW test scenario.
- The package contains four vials sealed in plastic containers and two jars sealed in plastic wrap. The samples are an organic solvent, liquid, and sand with their corresponding blanks. They are labeled:

Samples:

The three samples coded are as follows:

- O: Sample from container "Organic Waste"
- L: Sample appearing to be an organic liquid
- S: Soil from the facility's chemical loading/unloading area

with their corresponding blanks

OB

LB

SB

Your laboratory code is a two-digit number, which has been selected at random.

3. Please confirm the arrival date of the samples at your laboratory and their condition, by fax or email to:

Mr. Stefan Mogl, OPCW Laboratory (Fax:+31 15 2840679, email: OPCWrij@worldonline.nl)

Regards,		
Armando Alcaraz		

OPCW

Annex C: Dispatch Notification

The following is an example of e-mail or fax notification.

Dear Prof. Col József Fürész and Lt Gellért Karvaly,

Below is the web address and FedEx tracking number for your OPCW test samples:

http://www.fedex.com/cgibin/tracking?action=track&language=english&cntry_code=us&initial=x&t racknumbers=40090164874

FedEx Track numbers = 40090164874

Please acknowledge the receipt of the fax or email to:

Fax: (925) 423-9014 Email: <u>alcaraz1@llnl.gov</u>

Best regards, Armando Alcaraz Program Element Leader Lawrence Livermore National Laboratory Forensic Science Center, L-178 7000 East Ave Livermore, CA 94550-9234 (925) 423-6889 phone (925) 423-9014 fax **Annex D: Sample Receipt**

Country	Name of laboratory and address	Air Freight Carrier	Airway bill number	Receipt package date
Austria	RD-ARWT/ABCUT Vorgartenstr. 225 A-1024 Wien Austria	FedEx	40090164815	13/10/2004
Belgium	DLD (Departement Laboratoria van Defensie) Kwartier Majoor Housiau Martelarenstraat 181 B-1800 Vilvoorde (Peutie) Belgium	FedEx	40090164841	12/10/2004
Czech Republic	Research Institute for Organic Syntheses Rybitvi 296 532 18 Pardubice 20 Czech Republic	FedEx	40090164852	12/10/2004
Denmark	Ministry of Defence, Emergency Management Agency, Chemical Division Universitetparken 2, Nørre Alle 67, 7 th floor DK-2100 Copenhagen Denmark	FedEx	4009016505025	12/10/2004
Finland	Finnish Institute for Verification of the Chemical Weapons Convention (VERIFIN) P.O. Box 55 00014 University of Helsinki A.I.Virtasen aukio 1, 00560 Helsinki Finland	Air France	057-4146 8000	21/10/2004
France	Centre d'Etudes du Bouchet (CEB) Section Analyses Chimiques PO Box 3 91710 Vert-le-Petit France	FedEx	40090164863	11/10/2004

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Hungary	Department of Toxicology Institute of Health Protection Hungarian Defence Forces 44 Róbert károly körút, Budapest, H-1134 P.O.Box 68, Budapest, H1555 Hungary	FedEx	40090164874	12/10/2004			
India-IICT	Indian Institute of Chemical Technology Analytical Division Tarnaka Hyderabad-500 007 Andhra Pradesh India	Emirates Airlines	176-7000 2936	19/10/2004			
India - VERTOX	Defence Research & Development Establishment VERTOX Laboratory Jhansi Road Gwalior 474002 India	Cathay Pacific Airways	160-3462 0622	19/10/2004			
Malaysia	Department of chemistry Malaysia Ministry of Science, Technology and Innovation Jalan Sultan 46661 Petaling Jaya Malaysia	Cathay Pacific Airways	160-3462 0611	15/10/2004			
Morocco	Laboratoire Officiel d'Analyses et de Recherches Chimiques (LOARC) 25, Rue Nichakra Rahal 20 000 Casablanca Morocco	Air France	057-4146 79996	10/10/2004*			
Netherlands	TNO Prins Mauris Laboratory Lange Kleiweg 137 2288 GJ Rijswijk The Netherlands	FedEx	40090164911	12/10/2004			
Romania	NBC & Ecological Defence Scientific Research Centre Sos. Oltenitei 225, sector 4 RO-75 6872 BUCHAREST Romania	Air France	057-4146 8011	01/11/2004			

OPCW		Sai	mpie Prep, Part I, P	'age no. <u>20</u>
Russia CAL	Central Chemical Weapons Destruction Analytical Laboratory GosNIIOKhT (CAL) Shosse Entusiastov, 23 111024 Moscow Russian Federation	Air France	057-4146 8022	27/11/2004*
Spain	Fábrica Nacional "La Marañosa", Carretera San Martin de la Vega. Km. 10.5 San Martin de la Vega Madrid 28330 Spain	FedEx	40090164966	15/10/2004
Sweden	Swedish Defence Research Agency (FOI) Division of NBC Defence Cementvägen 20 SE-901 82 UMEÅ Sweden	FedEx	40090164970	14/10/2004
Ukraine	State Analytical Laboratory 61024 Kharkiv Petrovskogo St. 28 Ukraine	Lufthanza Cargo	020-7274 8712	01/11/2004*
UK-DSTL	Defence Science and Technology Laboratory, Porton Down, Salisbury Wiltshire, England SP4 0JQ	FedEx	40090164981 40090164756	11/10/2004 11/10/2004
OPCW Laboratory	OPCW Laboratory Heulweg 28-30 2288 GN Rijswijk The Netherlands	FedEx	40090165003 40090164760	12/11/2004 12/11/2004

^{*} This date represents the date when the Proficiency Test samples arrived at the point-of-entry only. These laboratories did not respond to the preparation laboratory, so the date (if any) of receipt of the samples at their facility is unknown.



ORGANISATION FOR THE PROHIBITION OF CHEMICAL WEAPONS

Report of the Sixteenth Official OPCW Proficiency Test

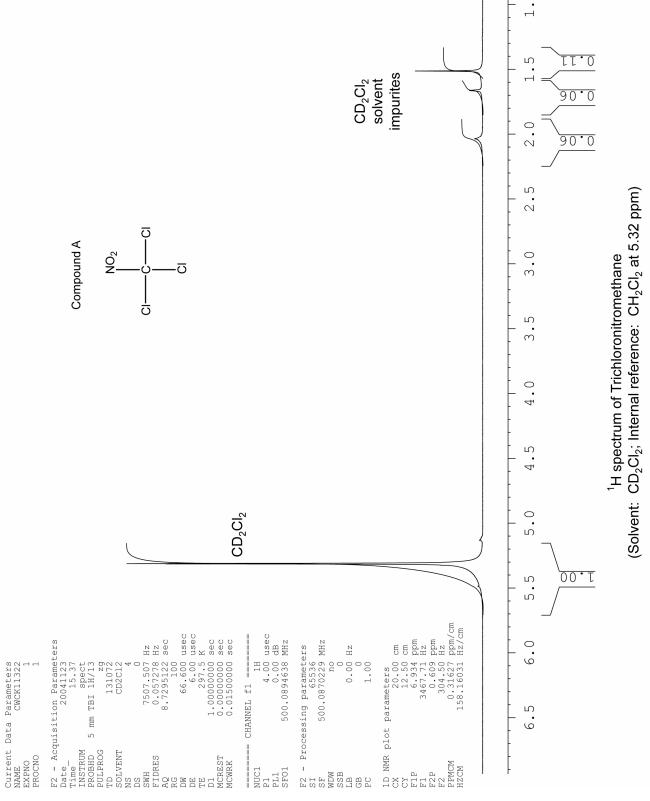
Part II: Purity Checks

Laboratory code: Sample Prep Lab

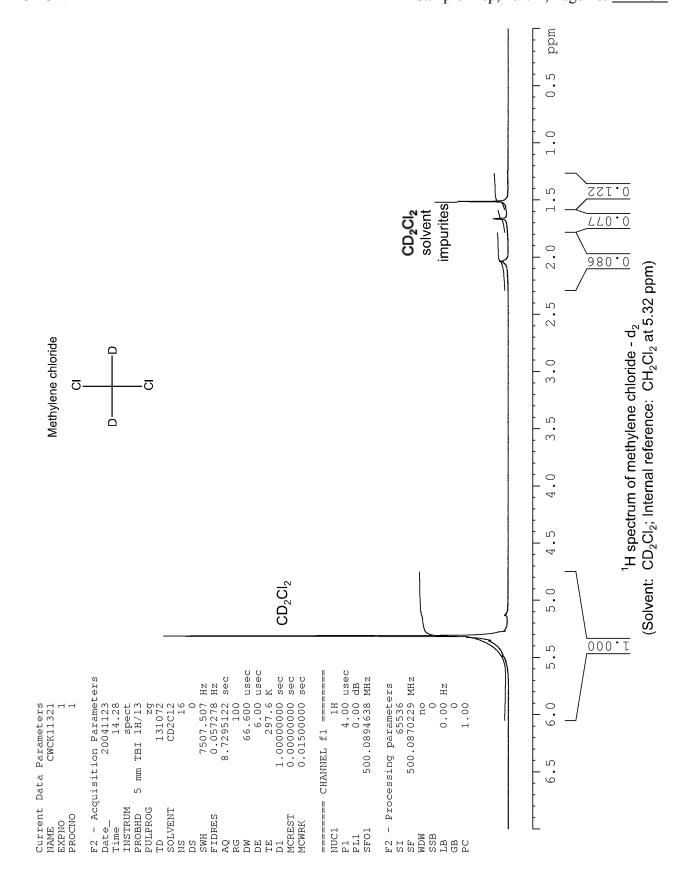
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Analytical technique 1: NMR ¹ H Observation	ompound number H	
Analytical technique 2: NMR ¹³ C { ¹ H } Observation		
Analytical technique 1: NMR ¹ H Observation	Analytical technique 2: NMR ¹³ C { ¹ H} Observation	
Analytical technique 1: NMR ¹ H Observation	ompound number I	
A district the annual localism of	Analytical technique 1: NMR ¹ H Observation	
Analytical technique 2: NMR "C{'H} Observation	Analytical technique 2: NMR ¹³ C{ ¹ H} Observation	

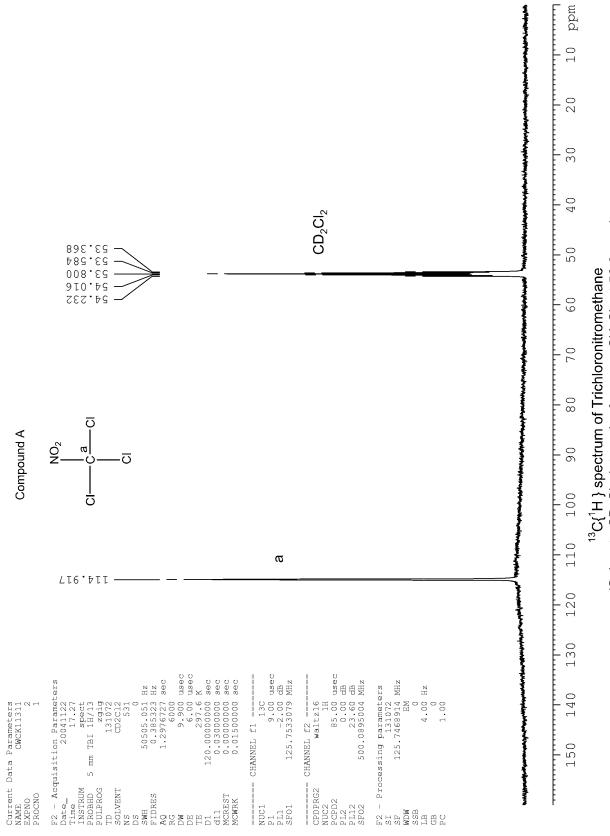
Laboratory code:	Prep Lab	Samp	le code	(s):	Purity (<u>Check</u>	Compou	and nu	mber:	<u>A</u>
Aliquot codes: (CW-CK-1-	132-2								
	hloronitro		in CD	2Cl2						
				Z - Z						
Compound stru	cture:	1	10_2							
		CI—C	ģ-—ή	CI						
		(ĊI							
NMR Method n	ame:	¹ H NMI	R							
METHOD DE	METHOD DESCRIPTION									
Instrument		Bruker I	ORX 5	500						
Manufacturer a	nd Type:	nd Type:								
Frequency:	V 1			Temp	erature	control	unit:	⊠ Ye	es 🗌 No	
Probe head: TBI				Temp	erature	:		26.5	°C	
Sample tube dia	meter:	5 mm	Solvent:				CD ₂ C	$\mathbb{C}l_2$		
pH of:		Sample	= Blank =				Refer	rence =		
δ reference reag	gent in San	iple:	⊠ In	ternal =	= CD ₂ C	l_2	☐ Exte	rnal =		
δ reference reag	gent in Ref	erence:	☐ Internal = ☐ External =							
ANALYCIC										
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Compared to lib			Sourc)		ther:	
Standard additio		1.	Sourc						iller.	
Spectral interpre					Buhlmann	, C. Affolt	ter, Struct	ure Det	termination	of
			Organic Compounds: Tables of Spectral Data, (Berlin: Springer-							
			Verlag) 2000.							
Experiments:	⊠ ¹H	$] ^{13}C\{^{1}H\} $] ¹⁹ F	∐ ³¹ P	{ ¹ H}	\square ³¹ P		COSY	
Assignment(s):	Other:	Chemica	l chift((c)			Coun	ling co	nstant(s)	
Assignment(s):	Sample sp			rence spo	ectrum	Sample	spectrum		Reference sp	ectrum
	[ppm]		Ittici	[ppm]	cer um	-	speed din Hz]	` ``	Hz]	ecti um
T	TD1		41.	1 4	11 1 .	lu an co			1 1	, ,
Interpretative comments	There are no its impuritie									
	performed v						50170	,· I a	-13, throng	



Laboratory code:	Prep Lab	Samp	le code	(s):	Purity (<u>Check</u>	ck Compound number: (
Aliquot codes:	CW-CK-1-	132-1								
	ent backgr		2Cl ₂							
Compound stru	cture:	D	 D	,						
		Ċ	l							
NMR Method n	ame:	¹ H NMI	R							
METHOD DE	SCRIPT	ION								
Instrument		Bruker I	ORX 5	500						
Manufacturer a	nd Type:									
Frequency:		500.09 N	MHz Temperature control unit:							
Probe head:		TBI		Temp	erature	2:		26.5 °C		
Sample tube dia	ımeter:	5 mm		Solve	nt:			CD ₂ Cl ₂		
pH of:		Sample	=	= Blank =				Reference =		
δ reference reas	gent in San	ıple:	⊠ In	ternal =	CD ₂ C	l_2	Exte	rnal =		
δ reference reag	gent in Ref	erence:	☐ Internal = ☐ External =							
ANALYSIS	,									
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Compared to lib		1:	Sourc		OCAD	(Code)	Other:		
Standard addition			Sourc		hlmone	C Affalt	or Ctrost	ava Datarminatia	un of	
Spectral interpre	tation:		E.Pretsch, P. Buhlmann, C. Affolter, <u>Structure Determination of Organic Compounds: Tables of Spectral Data</u> , (Berlin: Springer-Verlag) 2000.							
Experiments:	∐ H ☐ Other:] ¹³ C{ ¹ H}] ¹⁹ F	☐ ³¹ P	{ ¹ H}	31P	☐ COSY		
Assignment(s):		Chemica		,				ling constant(s)		
	Sample sp [ppr		Refei	ence spo [ppm]	ectrum	Sample s	-	n Reference [H	-	
Interpretative comments	Background	¹ H NMR o	of solve	nt confir	ming sol	vent impuri	ties shov	vn in ¹ H NMR of	sample A.	



Laboratory code:	Prep Lab	Samp	Sample code(s):		Purity (Check Compo		und number:		<u>A</u>	
Aliquot codes: CW-CK-1-131-1											
Sample: Trichloronitromethane in CD ₂ Cl ₂											
F											
Compound structure:		NO_2									
			2								
		CI——Ç ^a —CI									
		ĊI									
NMR Method name:		¹³ C{ ¹ H}	NMF	₹							
METHOD DESCRIPTION Instrument Bruker DRX 500											
Instrument Manufacturer and True		Bruker I	JKX :	500							
Manufacturer and Type:		125.75 MHz Ten			orotur	aontrol	unit.	⊠ Yes □ No			
Frequency: Probe head:		TBI		_	Temperature control unit:						
Sample tube diameter:		5 mm		Temperature: Solvent:				CH ₂ Cl ₂ /CD ₂ Cl ₂			
pH of:		Sample		Blank =			Reference =				
-	4 • 0	<u> </u>					□ E4-	rnal =			
δ reference reag											
δ reference reag	gent in Ref	erence:	erence: Internal =					External =			
ANALYSIS											
Compared to ref	cal:	Sourc	e: [Own Synthesis			Commercial				
Compared to library spectrum			Sourc	e:	OCAD (Code))	Other:			
Standard additio	Source:										
Spectral interpretation:		E.Pretsch, P. Buhlmann, C. Affolter, <u>Structure Determination of</u>									
	Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000.										
Experiments:	\Box ¹ H	¹³ C{¹H}	Veria] ¹⁹ F	□ ³¹ P	{ ¹ H}	□ ³¹ P		COSY		
I	Other:										
Assignment(s):		al shift(s)						ng constant(s)			
	Sample spectrum			Reference spectrum			spectrum	Reference spectrum [Hz]		ectrum	
[ppm] a 114.92 ppm		_	[ppm]	omj		[Hz]		[nz]			
a	114.92 ppm										
Interpretative	Purity = 1	<u></u>									
comments	i urity – r	00/0									
Comments											

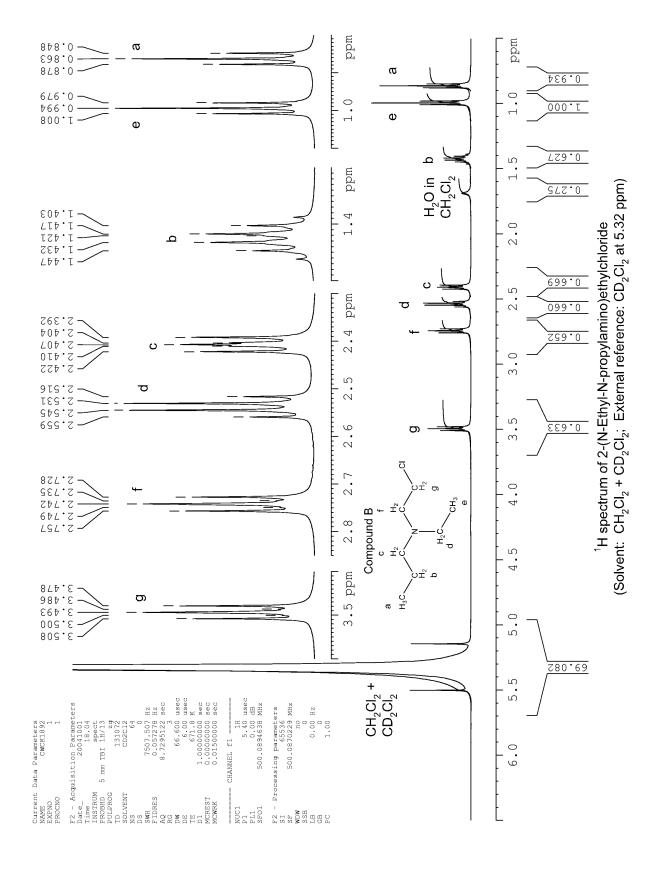


Solvent: CD_2Cl_2 , Internal reference: CH_2Cl_2 at 53.8 ppm)

comments

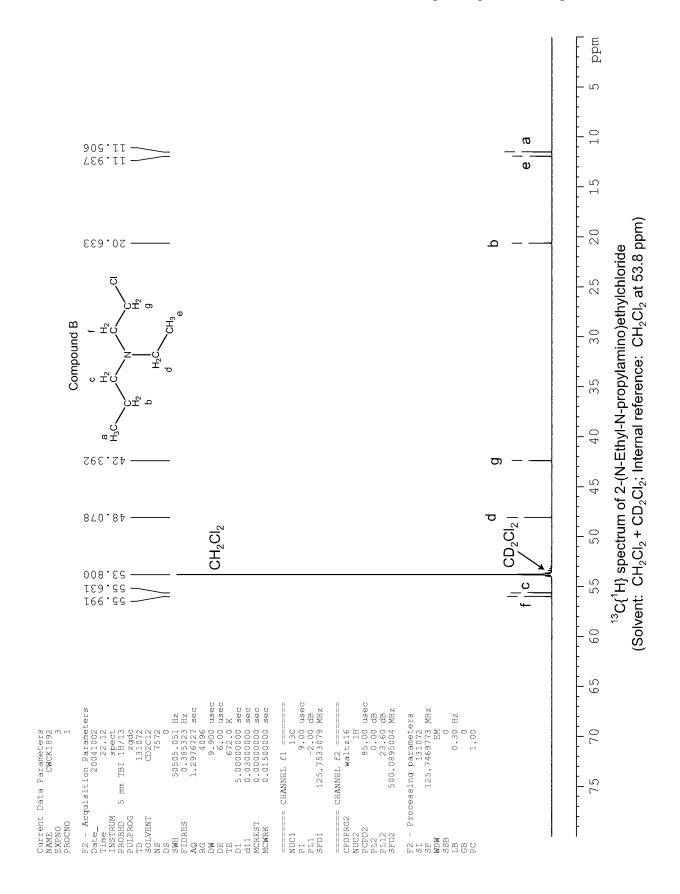
NMR METHOD AND ANALYSIS RESULTS

Compound number: Laboratory code: Prep Lab Sample code(s): **Purity Check** B Aliquot codes: CW-CK-1-89-2 2-(N-Ethyl-N-propylamino)ethylchloride in CH₂Cl₂/CD₂Cl₂ Sample: f **Compound structure:** С а H_2 H_2 H_3C CI H₂ H₂ b g H₂C ¹H NMR **NMR Method name:** METHOD DESCRIPTION Bruker DRX 500 **Instrument Manufacturer and Type:** \square No 500.09 MHz **Temperature control unit:** ⊠ Yes **Frequency:** Probe head: TBI 26.5 °C **Temperature:** Sample tube diameter: 5 mm **Solvent:** CH₂Cl₂/CD₂Cl₂ Sample = Reference = pH of: Blank = \boxtimes Internal = CD_2Cl_2 \square External = δ reference reagent in Sample: \square Internal = ∃External = δ reference reagent in Reference: **ANALYSIS** Compared to reference chemical: Source: Own Synthesis Commercial Compared to library spectrum: OCAD (Code Source: Other: Standard addition: Source: Spectral interpretation: E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000. \square ¹H 13C{¹H} 19 F $13^{1}P\{^{1}H\}$ COSY **Experiments:** Other: **Assignment(s):** Chemical shift(s) Coupling constant(s) Sample spectrum Reference spectrum Sample spectrum Reference spectrum [ppm] [ppm] [Hz] [Hz] 0.86 7.5 (b,a) a 0.99 7.1 (d,e)e 1.42 7.5 (a,b; c,b) b c 2.41 7.5(b,c)2.53 7.1 (e,d) d f 2.74 7.5 (g,f) 3.49 7.5 (f,g)Purity = 99.96% Interpretative

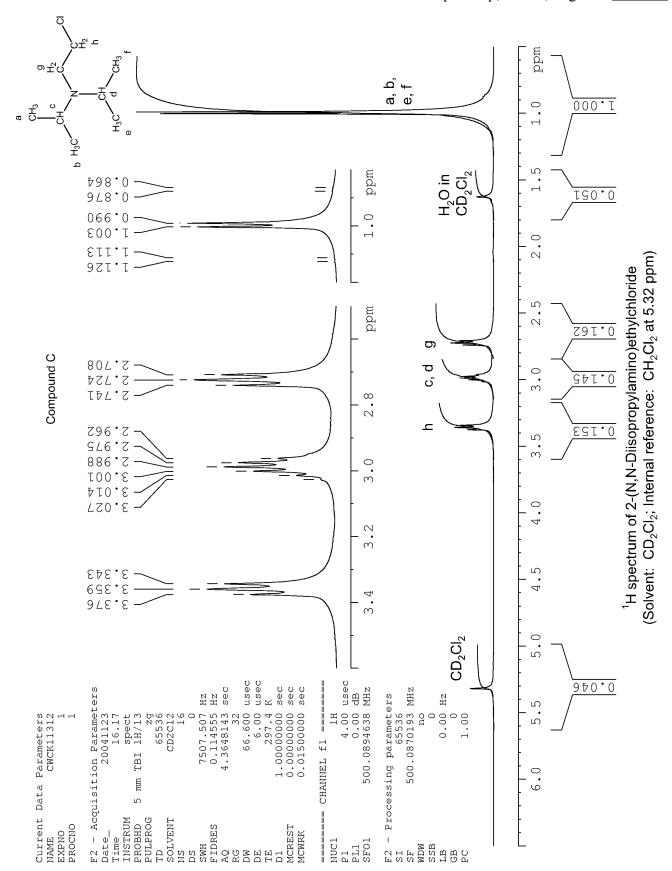


OPCW

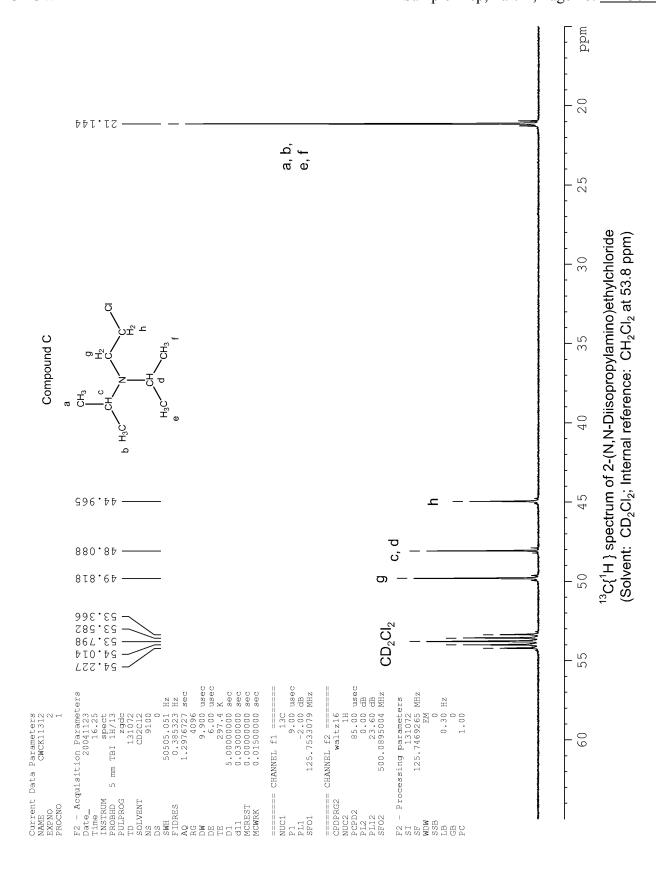
Laboratory code:	Prep Lab	<u>Prep Lab</u> Samp		le code(s): Purity (Check (Compour	nd number:	<u>B</u>	
Aliquot codes: (CW-CK-1-	89-2								
Sample: 2-(N-Ethyl-N-propylamino)ethylchloride in CH ₂ Cl ₂ /CD ₂ Cl ₂										
<u>C</u> 1.4	1				£					
Compound stru	cture:	а	C H.		f Ha					
		H_2 H_2 H_3 C C C								
		$\begin{array}{c ccccccccccccccccccccccccccccccccccc$								
		$\begin{bmatrix} & & & & & & & & & & & & & & & & & & &$								
	H_2C									
	a `CH ₃ e									
NMR Method n	ame:	¹³ C{ ¹ H}	NMF	₹						
METHOD DE	CCDIDE	ION								
METHOD DE	SCRIPT		. D. T. 7	-00						
Instrument	Bruker I	ORX 5	500							
Manufacturer a	105.75	47.7	TD.			•,				
Frequency:	125.75 MHz				e control u		Yes □ No			
Probe head:		TBI		Temperature:				26.5 °C		
Sample tube dia	5 mm		Solvent:				CH ₂ Cl ₂ /CD ₂ Cl ₂			
pH of:		Sample =		Blank =]	Reference =		
δ reference reagent in San		iple: \square Internal = CD_2Cl_2 \square Extended						nal =		
δ reference reag	gent in Ref	erence:] External =				
ANALYSIS										
Compared to reference chemi				e:	Own S	ynthesis		Commercial		
Compared to lib	:	Sourc		_ ` /			Other:			
Standard addition:		Source:						D : ::		
Spectral interpretation:		E.Pretsch, P. Buhlmann, C. Affolter, <u>Structure Determination of Organic Compounds</u> : <u>Tables of Spectral Data</u> , (Berlin: Springer-								
		Verlag) 2000.							gcı-	
Experiments:	☐ ¹H ☐ Other:	$I^{13}C\{^{1}H\}$								
Assignment(s):		Chemica	al shift(s)			Coupling constant(s)				
	Sample sp		Reference spectrum			Sample sp	ectrum	Reference spectrum		
	[ppm]		[ppm]			[Hz]		[Hz]		
a	11.51 11.94									
e b	20.63									
g	42.39									
d	48.08									
c	55.63									
f	55.99								·	
Interpretative comments										



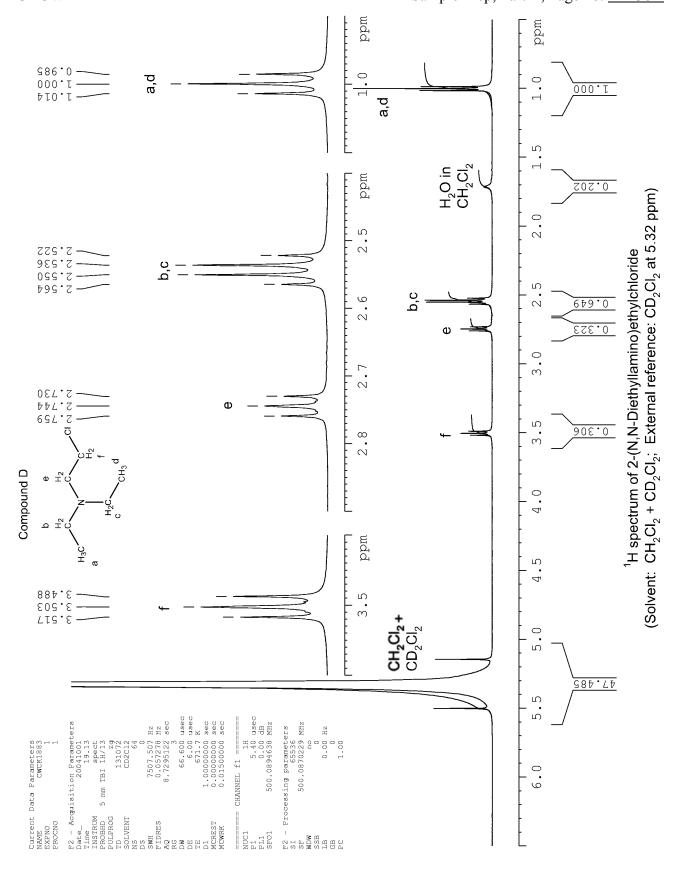
		~								
Laboratory code:	Prep Lab	Samp	le code	(s): <u>Purity (</u>	<u>Check</u> Compo	ound nu	mber: <u>C</u>			
Aliquot codes: CW-CK-1-131-2										
Sample: 2-(N,N-diisopropylamino)ethylchloride in CD ₂ Cl ₂										
Compound structures a										
Compound structure:			a CH ₃							
			1 *	g H ₂						
		CH C CI								
		b H ₃ C N C H ₂								
	CH ₂ h									
	H ₃ C d CH ₃									
e f										
NMR Method n	name:	¹ H NM	R							
METHOD DESCRIPTION										
Instrument		Bruker DRX 500								
Manufacturer a										
Frequency:		500.08 MHz		Temperature	control unit:	⊠ Y€	Yes No			
Probe head:		TBI		Temperature	2.	26.5 °C				
Sample tube diameter:		5 mm		Solvent:		CD ₂ Cl ₂				
pH of:		Sample	=	Blan	$\mathbf{k} =$	Reference =				
δ reference reas	gent in San	nple:	⊠ In	ternal = $\mathbf{C}\mathbf{D_2}\mathbf{C}$	l ₂ Ext	ernal =				
δ reference reas	gent in Ref	erence:	☐ In	ternal =	☐ External =					
ANALYSIS										
Compared to reference chemi		cal:	Sourc	e: Own S	ynthesis	☐ Co	ommercial			
Compared to library spectrum		` , ,					her:			
Standard addition		Source:								
Spectral interpretation:		E.Pretsch, P. Buhlmann, C. Affolter, <u>Structure Determination of Organic Compounds</u> : <u>Tables of Spectral Data</u> , (Berlin: Springer-								
		Verlag) 2000.								
Experiments:	\square ¹ H] ¹³ C{ ¹ H}			$\{^1H\}$ \square ^{31}P		COSY			
	Other:									
Assignment(s):	Chemic		,	. /			constant(s)			
	Sample spectrum		Reference spectrum [ppm]		Sample spectr [Hz]	um	Reference spectrum [Hz]			
	[ppm]		լբբույ		[112]		[IIL]			
lahef						f;d)				
a,b,e,f	1.00, 0.99				6.5 (a,c; b,c; e,d;	f;d)				
g	1.00, 0.99 2.72									
g c,d	1.00, 0.99 2.72 2.99				6.5 (a,c; b,c; e,d; 8.1 (g,h)					
g c,d h	1.00, 0.99 2.72 2.99 3.36				6.5 (a,c; b,c; e,d; 8.1 (g,h) 6.5 (c,a; c,b; d,e;					
g c,d	1.00, 0.99 2.72 2.99				6.5 (a,c; b,c; e,d; 8.1 (g,h) 6.5 (c,a; c,b; d,e;					



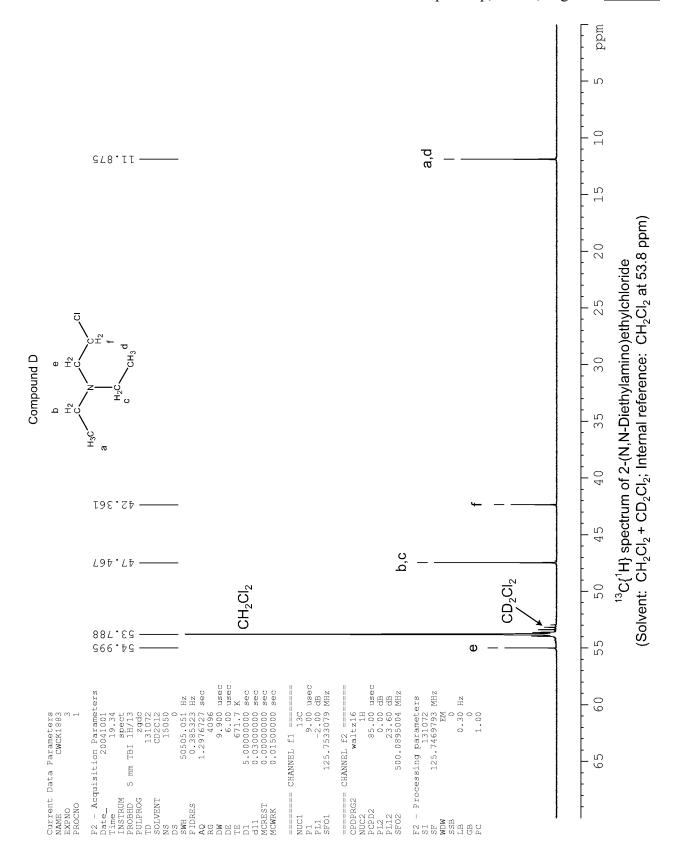
Laboratory code:	Prep Lab	Sampl	le code	(s):	Purity C	<u>Check</u>	Compou	ınd number:	<u>C</u>
Aliquot codes:	CW-CK-1-	131-2							
Sample: 2-(N	,N-diisopro	pylamin	o)ethy	ylchlor	ide in C	CD ₂ Cl ₂			
		T							
Compound stru	ecture:	(CH ₃ c H CH _d	g H ₂ C C H ₃					
NMR Method n	ame:	¹³ C{ ¹ H}	NMF	R					
METHOD DE	SCRIPT								
Instrument		Bruker I	ORX 5	500					
Manufacturer a	ınd Type:								
Frequency:		125.75 N	ИHz	-		control	unit:	Yes N	0
Probe head:			TBI Temperature: 26.5 °C						
Sample tube dia	ameter:	5 mm		Solve				CH ₂ Cl ₂ /CD ₂ C	
pH of:		Sample			Blan			Reference =	
δ reference reag					= CD ₂ C	l ₂		rnal =	
δ reference reag	gent in Ref	erence:	∐ In	ternal =	=		Exte	rnal =	
ANALYSIS				_	_				
Compared to ref			Sourc			ynthesis		Commercial	
Compared to lib Standard additio		1:	Sourc	_	OCAD	(Code)	Other:	
Spectral interpre					Ruhlmann	C Affolto	er Struct	ure Determination	n of
Spectral interpre	tution.		Organ	nic Comp		•		ata, (Berlin: Spri	
		7 12 .1 .		g) 2000.	- 21	.1	21		
Experiments:	☐ ¹H ☐ Other:	¹³ C{¹H}		」¹³F	31P	{'H} [☐ ³¹ P	☐ COSY	
Assignment(s):	Other.	Chemica	l shift((s)			Coup	ling constant(s)	
	Sample sp		Refe	rence sp		Sample s	-		
1 0	[pp1	n]		[ppm]		[H	[z]	[Hz	<u>[</u>
a,b,e,f	21.14								
h	44.96								
c,d	48.09								
g	49.82								
Interpretative comments									



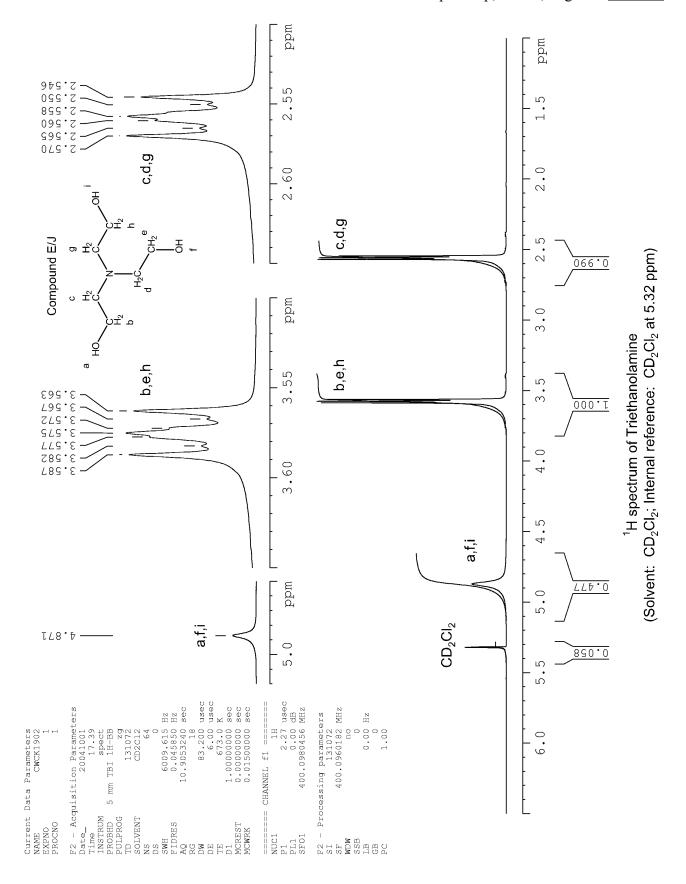
<u> </u>		НОД							
Laboratory code:	Prep Lab	Sampl	e code((s):	Purity (<u>Check</u>	Compou	ınd number:	<u>D</u>
Aliquot codes: C	CW-CK-1-	88-3							
Sample: 2-(N,	N-Diethyl:	amino)etl	ıylchl	oride in	n CH ₂ C	Cl_2/CD_2C	$\mathbb{C}\mathbf{l_2}$		
Compound struc	cture:	H₃C	b H ₂ C	e H ₂ C N	C H ₂	_CI			
		a f f c CH ₃ d							
NMR Method na	ame:	¹ H NMF	₹						
METHOD DES	SCRIPT	ION							
Instrument Manufacturer a	nd Type:	Bruker I	ORX 5	500					
Frequency:		500.09 MHz Temperature control unit: ⊠ Yes □ No							
Probe head:		TBI Temperature: 26.5 °C							
Sample tube dia	meter:	5 mm		Solven	ıt:			CH ₂ Cl ₂ /CD ₂ C	
pH of:		Sample	=		Blan	k =		Reference =	
δ reference reag	ent in San	nple:	⊠ In	ternal =	CD ₂ C	$\overline{\mathbf{l}_2}$	☐ Exte	rnal =	
		-	☐ In	ternal =			— Exte	 rnal =	
ANALYSIS		δ reference reagent in Reference:							
								Commercial	
Compared to libra	ary spectrum		Sourc	e :)	Commercial Other:	
	ary spectrum 1:		Source Source E.Pret Organ	e: Esch, P. B	OCAD uhlmann	(Code n, C. Affol		=	
Compared to libra Standard addition	ary spectrum ation:	13C{1H	Source Source E.Pret Organ Verlag	e: e: tsch, P. B	OCAD uhlmanr ounds:	(Code n, C. Affol		Other: ure Determination ata, (Berlin: Sprin	
Compared to librate Standard addition Spectral interpret Experiments:	ary spectrum n: cation:	13C{ ¹ H	Source Source E.Prete Organ Verlag	e:e :e : _	OCAD uhlmanr ounds:	(Code n, C. Affol Tables of S	Spectral D 31P	Other: ure Determination ata, (Berlin: Sprin	nger-
Compared to libration Standard addition Spectral interpret	ary spectrum ation:	13C{1H	Source Source E.Pret Organ Verlas H}	e:e :e :	uhlmann ounds: 3	(Code n, C. Affol Tables of S $P{^1H}$	Spectral D 31P Coupli	Other: ure Determination ata, (Berlin: Sprin COSY ing constant(s)	nger-
Compared to librate Standard addition Spectral interpret Experiments:	ary spectrum n: ation: Yes H Other San	: 13C{1H :: Chemica	Source Source E.Prete Organe Verlage I}	e:e:e:e:e:e:e:e: _	uhlmannounds: 7	(Code n, C. Affol Tables of S P{¹H} Sai	Spectral D 31P Coupling mple	Other: ure Determination ata, (Berlin: Sprin COSY ing constant(s) Reference s	pectrum
Compared to librate Standard addition Standard addition Spectral interpret Experiments: Assignment(s):	ary spectrum ation:	: 13C{1H :: Chemica	Source Source E.Prete Organe Verlage I}	e:e :e :	uhlmannounds: 7	(Code n, C. Affol Tables of S P{¹H} San spectr	Spectral D 31P Coupling mple um [Hz]	Other: ure Determination ata, (Berlin: Sprin COSY ing constant(s) Reference s	pectrum
Compared to librate Standard addition Standard addition Spectral interpret Experiments: Assignment(s):	ary spectrum ation: All IH Other San spectrui	: 13C{1H :: Chemica	Source Source E.Prete Organe Verlage I}	e:e:e:e:e:e:e:e: _	uhlmannounds: 7	(Code n, C. Affol Tables of S P{¹H} San spectr 7.0 (b,a	Couplimple um [Hz]	Other: ure Determination ata, (Berlin: Sprin COSY ing constant(s) Reference s	pectrum
Compared to librate Standard addition Standard addition Spectral interpret Experiments: Assignment(s): a,d b,c e	ary spectrum ation: The standard of the spectrum ation:	: 13C{1H :: Chemica	Source Source E.Prete Organe Verlage I}	e:e:e:e:e:e:e:e: _	uhlmannounds: 7	(Code n, C. Affol Tables of S P{¹H} San spectr	Couplimple um [Hz] c; c,d) c; d,c)	Other: ure Determination ata, (Berlin: Sprin COSY ing constant(s) Reference s	pectrum
Compared to librate Standard addition Standard addition Spectral interpret Experiments: Assignment(s):	ary spectrum ation: The other spectrum 1.00 2.54	: 13C{1H :: Chemica	Source Source E.Prete Organe Verlage I}	e:e:e:e:e:e:e:e: _	uhlmannounds: 7	(Code n, C. Affol Fables of S P{ ¹ H} San spectro 7.0 (b,a) 7.0 (a,b)	Couplimple um [Hz] i; c,d) o; d,c)	Other: ure Determination ata, (Berlin: Sprin COSY ing constant(s) Reference s	pectrum



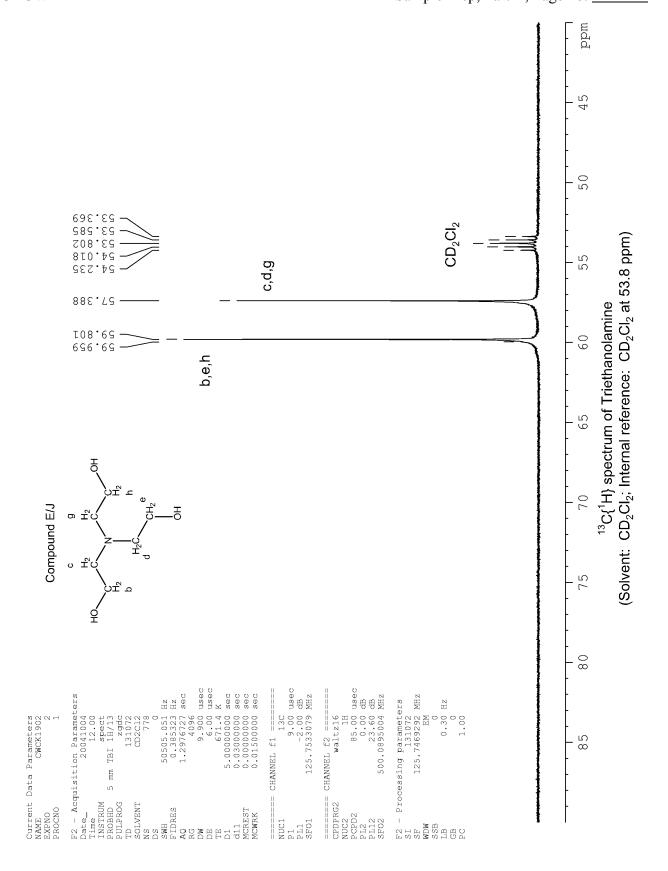
Laboratory code: Prep Lab Sample code(s): **Purity Check Compound number:** D Aliquot codes: CW-CK-1-88-3 2-(N,N-Diethylamino)ethylchloride in CH₂Cl₂/CD₂Cl₂ Sample: b **Compound structure:** е H_2 H_2 .CI H₃C \tilde{H}_2 а H₂C CH₃ ¹³C{¹H} NMR **NMR Method name:** METHOD DESCRIPTION Bruker DRX 500 Instrument **Manufacturer and Type:** 125.75 MHz \square No **Temperature control unit:** X Yes **Frequency:** Probe head: TBI 26.5 °C **Temperature:** Sample tube diameter: 5 mm **Solvent:** CH₂Cl₂/CD₂Cl₂ Sample = Reference = pH of: Blank = \boxtimes Internal = CD_2Cl_2 \square External = δ reference reagent in Sample: \square Internal = \exists External = δ reference reagent in Reference: **ANALYSIS** Compared to reference chemical: Source: Own Synthesis Commercial Compared to library spectrum: OCAD (Code Source: Other: Standard addition: Source: Spectral interpretation: E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000. □ ¹H \square ¹⁹F $\prod_{1}^{31} P\{^{1}H\}$ \square 31P \square ¹³C{¹H} **Experiments:** $\sqcap \cos y$ □ Other: **Assignment(s):** Chemical shift(s) **Coupling constant(s)** Sample Reference Sample **Reference spectrum** spectrum [ppm] spectrum [ppm] spectrum [Hz] [Hz] 11.87 a,d 42.36 b,c 47.47 54.99 **Interpretative** comments



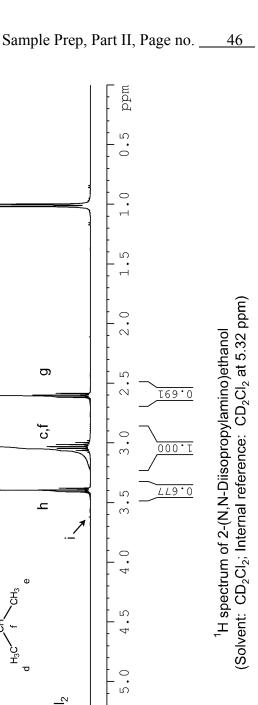
Laboratory code:	Prep Lab	Sampl	le code	(s): <u>Purity (</u>	<u>Check</u>	Compour	nd number:	<u>E, J</u>
Aliquot codes: C	W-CK-1-	90-2						
	hanolamir		Cl ₂					
•								
Compound struc	eture:	a HO C						
NMR Method na	ime:	¹ H NMI	₹					
METHOD DES								
Instrument		Bruker A	Avanc	e 400				
Manufacturer an	nd Type:							
Frequency:		400.10 N	ИHz	Temperature	e control	unit:	⊠ Yes □ No	
Probe head:		TBI Temperature: 26.5 °C						
Sample tube dia	meter:	5 mm		Solvent:			CD ₂ Cl ₂	
pH of:		Sample	=	Blan	k =		Reference =	
δ reference reage	ent in San	iple:	⊠ In	$ternal = CD_2C$	l_2	☐ Extern	nal =	
δ reference reage		_	□In	ternal =		Extern	nal =	
ANALYSIS								
Compared to refe	rence chemic	cal:	Sourc	e: Own S	ynthesis		Commercial	
Compared to libra	, i	:	Sourc	e: OCAD	(Code)	Other:	
Standard addition			Sourc		G + 60 1			C
Spectral interpreta			Organ Verla	nic Compounds: 7	<u>Γables of S</u>	Spectral Da	re Determination ta. (Berlin: Sprin	_
Experiments:	⊠ ¹H ☐ Other:			\Box ¹⁹ F \Box ³¹	P{¹H}	□ ³¹ P	COSY	
Assignment(s):		Chemic				•	ng constant(s)	
	Sam spectrun	-		Reference etrum [ppm]		nple um [Hz]	Reference s _j [Hz]	oectrum
c d a	2.56	ո [հհայ	spec	աս լբբույ	4.8 (b,c;			
c,d,g					4.8 (c,b;			
b,e,h	3.575				T.0 (C,0,	u,c, g,11 <i>)</i>		
a,f,i	4.87							
Interpretative comments	Purity = 9	99.7%						

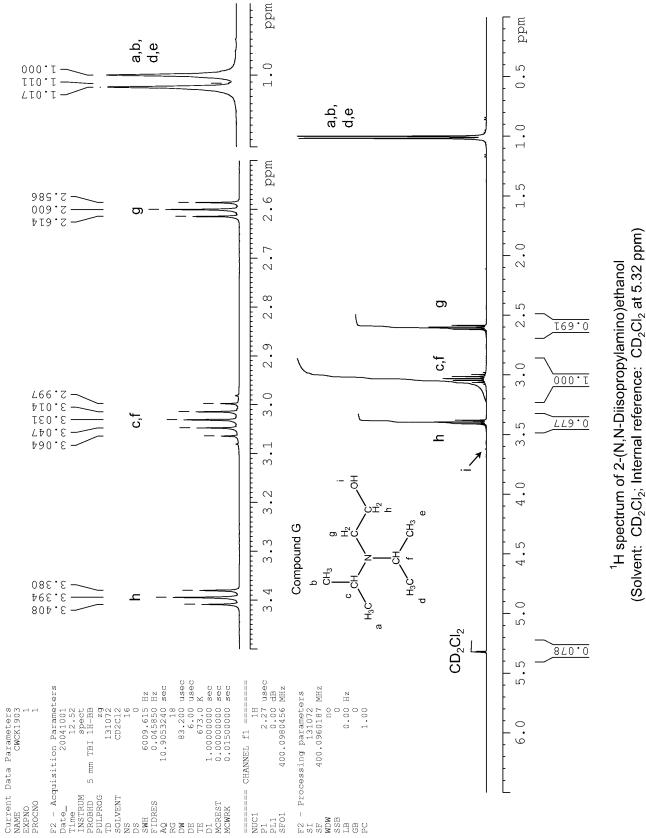


Laboratory code:	Prep Lab	Samp	le code	(s): <u>Pu</u>	ırity (<u>Check</u>	Compou	ınd number:	\mathbf{E}, \mathbf{J}
Aliquot codes: C	W-CK-1-	90-2							
_	hanolamii		Cl2						
			.012						
Compound struc	eture:	a HO CH ₂ b	HO C H_2 H_2 H_2 OH						
NMR Method na	ame:	¹³ C{ ¹ H}	NMF	₹					
METHOD DES	SCRIPT	ION							
Instrument		Bruker I	ORX 5	500					
Manufacturer an	nd Type:								
Frequency:		125.75 N	МHz	Temper	ature	control	unit:	⊠ Yes □] No
Probe head:		TBI	TBI Temperature: 26.5 °C						
Sample tube dia	meter:	5 mm		Solvent:				CH ₂ Cl ₂ /Cl	O ₂ Cl ₂
pH of:		Sample	=		Blan	$\mathbf{k} =$		Reference	=
δ reference reag	ent in San	nple:	⊠In	ternal = C	CD ₂ C	$\overline{\mathbf{l_2}}$	☐ Exte	rnal =	
δ reference reag	ent in Ref	erence:	□In	ternal =			☐ Exte	rnal =	
ANALYSIS									
Compared to refe			Sourc		•	ynthesis		Commerc	cial
Compared to libra		1:	Sourc		OCAD	(Code)	Other:	
Standard addition			Sourc		1	C Affal	ton Ctmast	Datamaina	4i.a., a.f.
Spectral interpret	ation:							ure Determina ata, (Berlin: S	
				g) 2000.	nas. I	uoles of E	респать.	utu, (Derim. E	pringer
Experiments:	☐ ¹ H ☐ Other:		I } [□ ¹⁹ F	³¹]	P{ ¹ H}	□ ³¹ P		Y
Assignment(s):		Chemic	al shif	ft(s)			Coupli	ing constan	t(s)
	Sam	-	F	Reference	;	Sar	nple	Referen	ce spectrum
	spectrun	n [ppm]	spec	etrum [pp	m]	spectri	um [Hz]		Hz]
c,d,g	57.39								
b,e,h	59.80								
Interpretative comments									

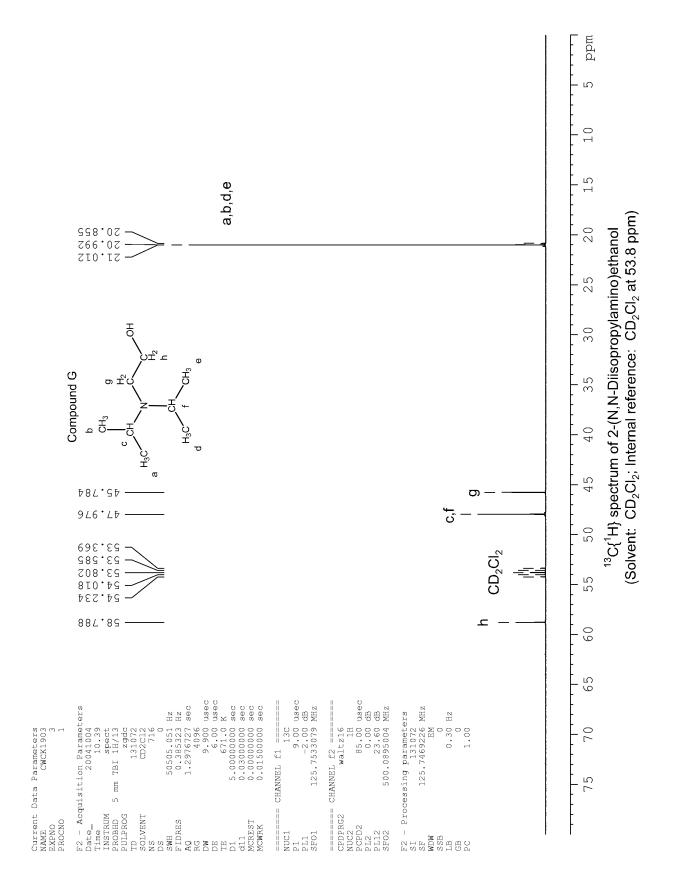


				•						
Laboratory code:	Prep Lab	Sampl	le code	(s): <u>Pu</u>	urity C	<u>Check</u>	Compou	ınd numk	er:	<u>G</u>
Aliquot codes: C	W-CK-1-	90-3								
	N-Diisopr		o)eth	anol in C	D ₂ Cl	2				
	, 21150p1	оруши	,		2201	<i>L</i>				
Compound struc	cture:	b								
-			CH ₃	g						
		c .C	Н	H ₂ .C.	.OH					
		H ₃ C N C								
		а	\mathbf{H}_2							
			CH "							
		H ₃ C	; t	CH₃ e						
NMR Method na	ame:	¹ H NMI	3							
METHOD DE	COLDE	LONI								
METHOD DES	SCRIPT									
Instrument		Bruker A	Avanc	e 400						
Manufacturer a	nd Type:									
Frequency:		400.10 MHz Temperature control unit: ⊠ Yes □ No								
Probe head:		TBI Temperature: 26.5 °C								
Sample tube dia	meter:	5 mm		Solvent:	:			CD_2Cl_2	2	
pH of:		Sample	=		Blan	$\mathbf{k} =$		Refere	nce =	
δ reference reag	ent in San	iple:		iternal = (CD_2C	l_2	Exte	rnal =		
δ reference reag	ent in Ref	erence:	☐ In	iternal =			☐ Exte	rnal =		
ANALYZONO										
ANALYSIS			G		2 0	.1 .			• •	
Compared to refe			Sourc			ynthesis			mercial	
Compared to libration Standard addition		l :	Sourc		JCAD	(Code)	Othe	r:	
Spectral interpret				tsch, P. Buh	ılmann	C Affol	ter Struct	ure Deter	mination of	f
Spectral interpret	ation.			nic Compou						_
			Verla	g) 2000.			•		1 0	
Experiments:	\boxtimes ¹ H	\square ¹³ C{ ¹ H	I } [□ ¹⁹ F	[31]	$P{^1H}$	□ ³¹ P	□С	OSY	
-	☐ Other:									
Assignment(s):		Chemica	al shif	ft(s)			Coupli	ing cons	stant(s)	
	Sam	ple	F	Reference	•	Sa	mple		Referen	ice
	spectrun	n [ppm]	spec	ctrum [pp	om]	spectr	um [Hz] s	pectrum	[Hz]
a,b,d,e	1.01						c,b; f,d; f,		-	
g	2.6					5.6 (h,g	(;)			
c,f	3.03						b,c; d,f; e,	f)		
h	3.39					5.6 (g,h	1)			
Interpretative	Purity = 9	99 8%			I	2.0 (B)I	-/			
comments										
	1									

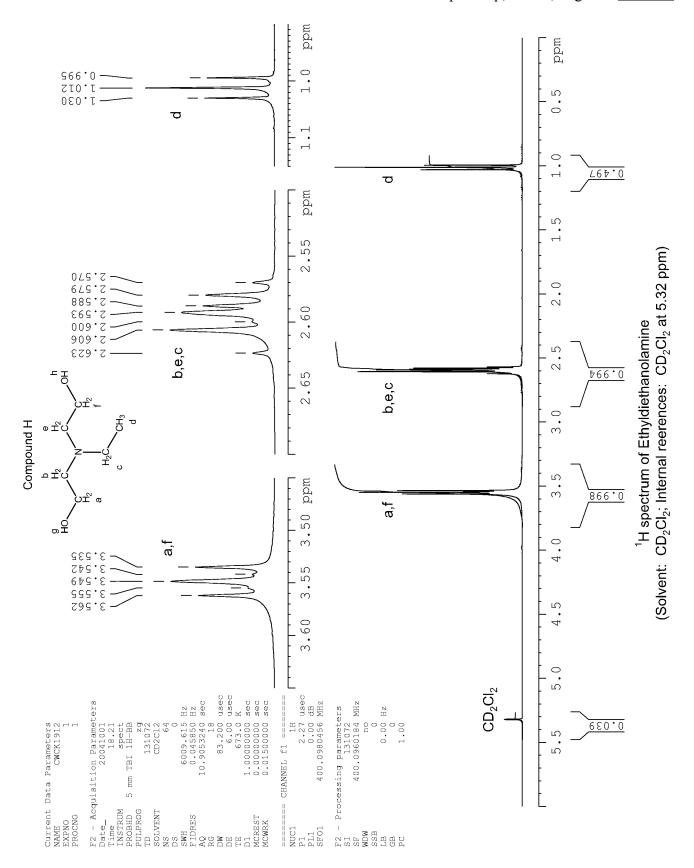




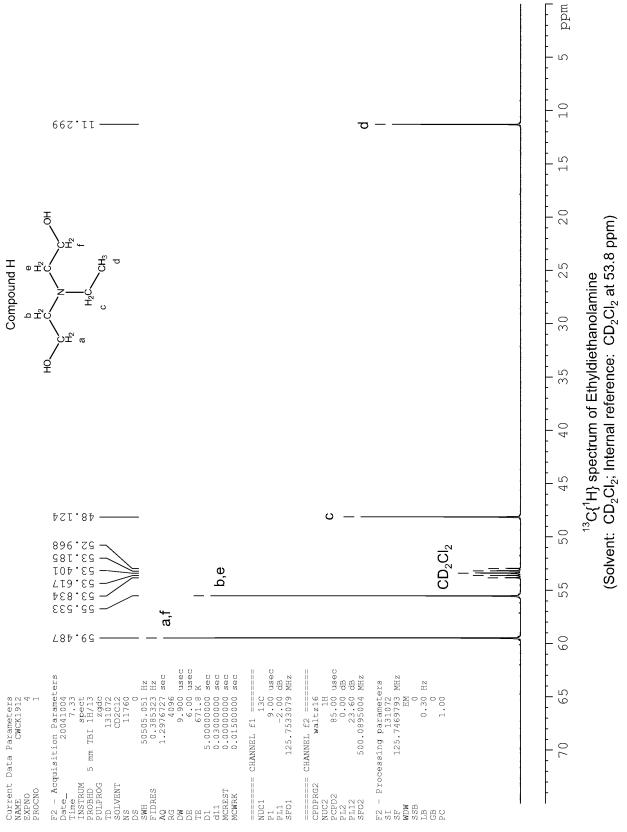
1 1111		1102	1111		1010	TLES					
Laboratory code:	Prep Lab	Samp	le code(s)	Purity (<u>Check</u>	Compoun	d number:	<u>G</u>			
Aliquot codes: C	W-CK-1-	90-3									
			o)ethan	ol in CD ₂ Cl	12						
	ТТВПБОРТ	орушин	i o je tii u i	or in eager	- 2						
Compound struc	cture:	b									
_		CH ₃ g									
		c .c	c H ₂ ' CH, CS, OH								
		H₃C	Ň	C H ₂							
		а		n ₂ h							
		H ₃ (CH C	CH ₃							
		d d	, , ,	e e							
NMR Method na	ame•	¹³ C{ ¹ H}	NMR								
THE PROPERTY OF THE		<u> </u>	1111111								
METHOD DES	SCRIPT	1									
Instrument		Bruker I	ORX 500)							
Manufacturer a	nd Type:						T				
Frequency:		125.75 N	MHz	Temperati		ol unit:	⊠ Yes [□ No			
Probe head:			TBI Temperature: 26.5 °C								
Sample tube dia	meter:	5 mm		Solvent:			CD ₂ Cl ₂				
pH of:		Sample	=	Blan	ık =	R	Reference =	=			
δ reference reag	ent in San	iple:		ternal = CD	₂ Cl ₂	☐ Exte	ernal =				
δ reference reag	ent in Ref	erence:		iternal =		☐ Exte	ernal =				
ANALYSIS											
Compared to refe	rence chemi	cal:	Source:		ynthesis		Commerci	al			
Compared to libr	• •	ı :	Source:	OCAD	(Code) [Other:				
Standard addition			Source :	D D 11	G + CC 1	<u> </u>	D : : :				
Spectral interpret	ation:			h, P. Buhlmanı Compounds: "							
			Verlag)		1 autes of 5	pectiai Dau	<u>a,</u> (Deriii. 5 ₁	minger-			
Experiments:	\Box ¹ H	\boxtimes ¹³ C{ ¹ H	ý		$P{}^{1}H}$	□ ³¹ P	□ COSY	-			
	Other		,		()						
Assignment(s):		Chemic	al shift(s	<u> </u>		Couplin	g constant	(s)			
9 (/	Sam			ference	San	nple		e spectrum			
	spectrun	-	spectr	um [ppm]		ım [Hz]		Hzj			
a,b,d,e	20.99	- 11 1		- 41 /		L J	L				
g	45.78							-			
c,f	47.98										
h	58.79										
Interpretative			I		ı		<u>I</u>				
comments											



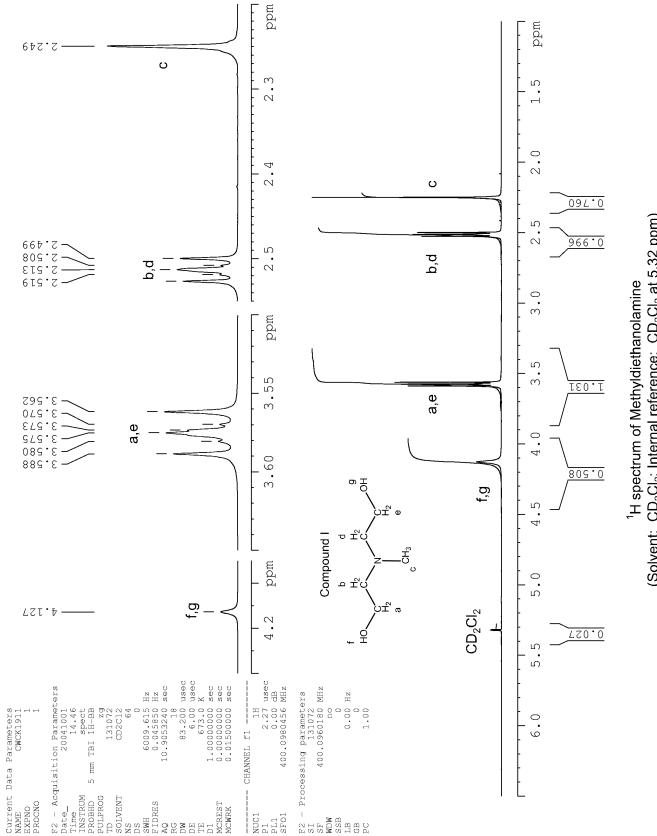
Laboratory code:	Prep Lab	Sample	code(s):	Purity C	hecl	<u>k</u> Comp	ound num	ber: <u>H</u>		
Aliquot codes: C	W-CK-1-9	91-2								
Sample: Ethyl	diethanola	mine in	CD ₂ Cl ₂							
Compound struc	cture:	g HO	b H ₂	e H ₂		h _OH				
		C H ₂ a	$\begin{array}{cccccccccccccccccccccccccccccccccccc$							
			c CH ₃							
		ler vo er								
NMR Method na	ame:	¹ H NMI	K							
METHOD DES	SCRIPTI									
Instrument		Bruker	Avance 4	400						
Manufacturer a	nd Type:									
Frequency:		400.10	MHz			re contr	ol unit:	⊠ Yes □ No		
Probe head:			TBI Temperature: 26.5 °C							
Sample tube dia	meter:	5 mm		Solvent				CD ₂ Cl ₂		
pH of:		Sample	<u>;</u> =	F	Blan	<u>k</u> =	<u> </u>	Reference =		
δ reference reag	ent in Sam	ple:		ral = CI	₂ Cl		☐ Extern	al =		
δ reference reag	ent in Refe	erence:	☐ Inter	mal =			Extern	al =		
ANALYSIS										
Compared to refe	rence chemic	al:	Source:	Ow	n Sy	nthesis		Commercial		
Compared to libra			Source:		AD	(Code) [Other:		
Standard addition			Source:	D D 11		G + 60 1:	G	D : :: :: :		
Spectral interpret	ation:			Compound				e <u>Determination of</u> a. (Berlin: Springer-		
Experiments:	\square ¹ H [\square Other:	□ ¹³ C{¹H	H} 🔲	¹⁹ F [] ³¹ F	? { ¹ H}	□ ³¹ P	□ COSY		
Assignment(s):		Chemica	al shift(s	5)			Couplin	g constant(s)		
	Sam	_		erence		Sam	_	Reference		
	spectrum	[ppm]	spectr	um [ppn	1]	spectru	m [Hz]	spectrum [Hz]		
d	1.01					7.2 (c,d)				
b,e,c	2.59									
a,f	3.55					5.6 (b,a;	e,f)			
Interpretative comments	Purity = 9	9.6%								
	l									



Laboratory code:	Prep Lab	Sampl	le code	(s): <u>l</u>	Purity (<u>Check</u>	Compoun	d number:	<u>H</u>
Aliquot codes: C	W-CK-1-	91-2							
	diethanol		CD ₂ C	Cl_2					
		T							
Compound struc	eture:	g HO C H ₂ a	HO C H_2 H_2 H_2 H_2 H_2						
NMR Method na	ame:	¹³ C{ ¹ H}	NMF	R					
METHOD DES	SCRIPT	ION							
Instrument		Bruker I	ORX 5	500					
Manufacturer ai	nd Type:								
Frequency:		125.75 N	ИHz	Tempe	rature	e control	unit:	⊠ Yes □ No	0
Probe head:		TBI		Tempe	rature	2:	2	26.5 °C	
Sample tube dia	meter:	5 mm		Solven	t:		(CD ₂ Cl ₂	
pH of:		Sample	=		Blan	k =	I	Reference =	
δ reference reage	ent in San	iple:	⊠ In	ternal =	CD ₂ C	l_2	☐ Extern	nal =	
δ reference reag	ent in Ref	erence:	☐ In	ternal =			☐ Extern	nal =	
ANALYSIS		_							
Compared to refe			Sourc			ynthesis		Commercial	
Compared to libra		1:	Sourc		OCAD	(Code)	Other:	
Standard addition Spectral interpret			Sourc		ıhlmarı	C Affeld	or Ctmot-	e Determination	of
Spectral interpret	ation.		Organ					<u>a.</u> (Berlin: Sprii	
Experiments:	☐ ¹H ☐ Other:	-			³¹]	P{ ¹ H}	□ ³¹ P	□ COSY	
Assignment(s):		Chemica	al shif	it(s)			Couplin	ng constant(s))
9 (7	Sam			Referenc	ee	Sar	nple	Reference s	
	spectrun	-		ctrum [p			ım [Hz]	[Hz	_
d	11.30					-	. 1	1	
С	48.12								
b,e	55.53								
a,f	59.49								
Interpretative comments								1	



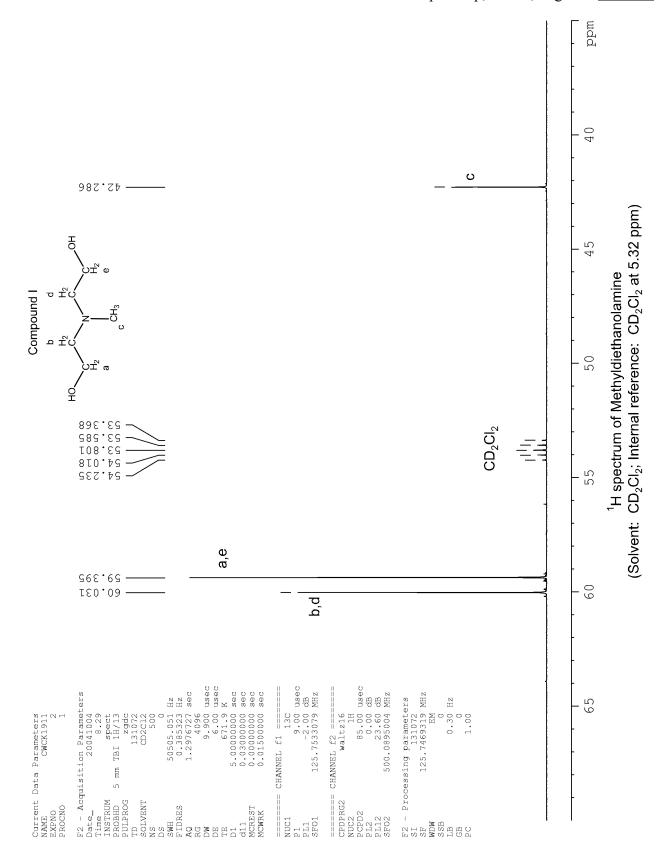
Laboratory code: Prep Lab Sample code(s): **Purity Check Compound number:** I Aliquot codes: CW-CK-1-91-1 Methyldiethanolamine in CD₂Cl₂ Sample: **Compound structure:** d b f H_2 g H_2 HO. OH C H₂ H_2 CH_3 ¹H NMR **NMR Method name:** METHOD DESCRIPTION Bruker Avance 400 Instrument **Manufacturer and Type:** \square No 400.10 MHz **Temperature control unit:** ⊠ Yes **Frequency:** Probe head: TBI 26.5 °C **Temperature:** Sample tube diameter: 5 mm **Solvent:** CD₂Cl₂ Sample = Blank =Reference = pH of: \boxtimes Internal = CD_2Cl_2 \square External = δ reference reagent in Sample: \square Internal = \exists External = δ reference reagent in Reference: **ANALYSIS** Compared to reference chemical: Source: Own Synthesis Commercial Compared to library spectrum: OCAD (Code Source: Other: Standard addition: Source: Spectral interpretation: E.Pretsch, P. Buhlmann, C. Affolter, Structure Determination of Organic Compounds: Tables of Spectral Data, (Berlin: Springer-Verlag) 2000. \Box ¹³C{¹H} \square ¹⁹F $\prod_{1}^{31} P\{^{1}H\}$ \square 31P \boxtimes ¹H **Experiments:** $\sqcap \cos y$ □ Other: **Assignment(s):** Chemical shift(s) **Coupling constant(s)** Sample Reference Sample **Reference spectrum** spectrum [ppm] spectrum [ppm] spectrum [Hz] [Hz] 2.25 c b,d 2.51 5.6 (a,b; d,e) 3.57 5.6 (b,a; e,d) a,e 4.13 f,gPurity = 99.7% **Interpretative** comments



(Solvent: CD₂Cl₂; Internal reference: CD₂Cl₂ at 5.32 ppm)

NIMD	METHOD	AND	ANAT	VCIC	DECIII TC
NVK	WIE I HUJIJ	AND	ANAI	1 X 212	KESULIS

Laboratory code:	<u>Prep Lab</u>	Samp	le code	(s): <u>l</u>	Purity C	<u>Check</u>	Compou	ınd number:	<u>I</u>
Aliquot codes: C	W-CK-1-	91-1							
Sample: Meth	yldiethan	olamine i	n CD	₂ Cl ₂					
* '	V	T	•						
Compound struc	cture:	_		b	d				
		f		H_2	H_2		g		
		HO	/	C_	C\		,OH		
			C_	, Ń.		`c_			
			\bar{H}_2			H_2			
			а	Ċŀ	- 12	е			
				c o	'3				
NMR Method na	ame•	¹³ C{ ¹ H}	NME	2					
TVIVIII IVICEIIOU III		<u> </u>	1 (11/11	<u> </u>					
METHOD DES	SCRIPT								
Instrument		Bruker I	ORX 5	500					
Manufacturer an	nd Type:			Ī					
Frequency:		125.75 N	МHz	Tempe	rature	e control	unit:	⊠ Yes [] No
Probe head:		TBI	TBI Temperature: 26.5 °C						
Sample tube dia	meter:	5 mm		Solven	t:			CD ₂ Cl ₂	
pH of:		Sample	=		Blan	$\mathbf{k} =$		Reference	<u>;</u> =
δ reference reag	ent in San	nple:	⊠ In	ternal =	CD ₂ C	l_2	☐ Exte	rnal =	
δ reference reag	ent in Ref	erence:	☐ In	ternal =			☐ Exte	rnal =	
ANALYSIS									
Compared to refe	rence chemi	cal:	Sourc	e:	Own S	ynthesis		Comme	rcial
Compared to libra		1:	Sourc	e:	OCAD	(Code)	Other:	
Standard addition			Sourc						
Spectral interpret	ation:							ure Determin	
				<u>11c Compo</u> g) 2000.	ounas: 1	ables of S	spectral D	ata, (Berlin:	Springer-
Experiments:	□ ¹H	\boxtimes ¹³ C{ ¹ F			☐ 31 ₁	р ∫¹ н ≀	□ ³¹ P		'V
Experiments.	Other		1) [Ц,	1 (11)	1		, 1
Assignment(s):	Other.	Chemica	al shif	ft(s)			Count	ing consta	nt(s)
rissignment(s).	Sam			Referenc	·e	Sar	nple		ice spectrum
	spectrun	_		ctrum [p			im [Hz]		[Hz]
С	42.29	ո լթթույ	spec	ti uiii [[piiij	spectr	ını [112]		[IIZ]
a,e	59.39								
b,d	60.03								
U,U	00.03								
Intownwatations									
Interpretative									
comments									





ORGANISATION FOR THE PROHIBITION OF CHEMICAL WEAPONS

Report of the Sixteenth Official OPCW Proficiency Test

Part III: Qualitative Analysis

Laboratory code: Sample Prep Lab

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OPCW

SUMMARY: PARTICIPATING LABORATORY

1. Participating laboratory

1. I al ticipating laborato	, i y
Laboratory code	Sample Prep Lab, aliquot #31
Name of the laboratory/institute participating in the test	Lawrence Livermore National Laboratory
Contact person	Mr. Armando Alcaraz
Address	PO Box 808, M/S L-178 7000 East Avenue Livermore, CA 94551
Telephone number	925-423-6889
Fax number	925-423-9014
Email address	Alcaraz1@llnl.gov

2. Analysts and authentication

	Name	Title	Pages*	Date**	Signature**
1	Mr. Armando Alcaraz	Principal Investigator	All	Nov. 30, 2004	
2	Dr. Hugh Gregg	Co-PI, Senior Chemist	All	Nov. 30, 2004	
3	Dr. Carolyn Koester	Research Scientist	Prep & GC/MS	Nov. 30, 2004	
4	Dr. Phil Pagoria	Research Scientist	Synthesis	Nov. 30, 2004	
5	Dr. Andrew Vance	Research Scientist	Synthesis	Nov. 30, 2004	
6	Dr. Robert Maxwell	Research Scientist	NMR analysis	Nov. 30, 2004	
7	Dr. Sarah Chinn	Research Scientist	NMR analysis	Nov. 30, 2004	
8	Mr. Rich Whipple	Scientist	Sample Prep	Nov. 30, 2004	
9	Ms. Tuijauna Mitchell-Hall	QA Manager	QA/QC	Nov. 30, 2004	
10					
11					
12					

^{*} Page numbers defining the responsibility area of the analyst; ** Date and signature of the responsible analyst;

SUMMARY: QUALITY ASSURANCE / QUALITY CONTROL (QA/QC)

. Status of the laboratory (tick where applicable)
Accreditation accepted: Year 2001 Accreditation body: American Association for Laboratory Accreditation Scope of accreditation: Chemical
Accreditation planned/pending: Target year Accreditation body: Scope of accreditation:
☐ Not accredited.
2. Quality system (tick where applicable)
 ✓ Described in a Quality Assurance Manual/Handbook. Quality system in accordance with: ☐ ISO 900
☐ No quality system. Please, fill in question number 3.
5. QA/QC Summary (Summary of the applied quality assurance and quality control (QA/QC) procedures concerning sample preparation, calibration, and analysis. Requested only from laboratories without a quality system).



THE AMERICAN ASSOCIATION FOR LABORATORY **ACCREDITATION**

ACCREDITED LABORATORY

A2LA has accredited

LAWRENCE LIVERMORE NATIONAL LABORATORY Livermore, CA

for technical competence in the field of

Chemical Testing

The accreditation covers the specific tests and types of tests listed on the agreed scope of accreditation. This laboratory meets the requirements of ISO/IEC 17025 -1999 "General Requirements for the Competence of Testing and Calibration Laboratories" and any additional program requirements in the identified field of testing.

Presented this 12th day of April 2004.



For the Accreditation Council Certificate Number 1914-01 Valid to February 28, 2006

For tests or types of tests to which this accreditation applies, please refer to the laboratory's Chemical Scope of Accreditation.



American Association for Laboratory Accreditation

SCOPE OF ACCREDITATION TO ISO/IEC 17025-1999

LAWRENCE LIVERMORE NATIONAL LABORATORY FORENSIC SCIENCE CENTER – OPCW PROJECT 7000 East Avenue Mailstop L-178 Livermore, CA 94550 Armando Alcaraz Phone: 925 423 6889

CHEMICAL

Valid To: February 28, 2006 Certificate Number: 1914-01

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following types of <u>Qualitative Tests for Chemicals related to Chemical Warfare Convention (CWC) in unknown samples:</u>

Sample Preparation	BB-SP5, BB-SP6, BB-SP8, BB-SP9,
	II CD1 II CD2 II CD2 II CD4

DE SI I, DE SI E, DE SI S, DE SI T

<u>Test</u>	Procedures
-------------	------------

Spectroscopy

Nuclear magnetic resonance BB-NMR1

Capillary zone electrophoresis / UV detection LL-CE1

Gas chromatography / Fourier Transform BB-IR1

Infrared Spectrometry

Chromatography

Gas chromatography / Element Specific Detectors BB-GC1

Gas chromatography / Mass Spectrometry BB-MS1

Liquid chromatography / Atmospheric pressure

Chemical Ionization / Mass Spectrometry BB-MS4

Liquid Chromatography / Electrospray Ionization

Mass Spectrometry LL-MS1

Chain of Custody for Laboratory LL-CC1

Work Instructions for the Preparation of Test Samples Q

for OPCW Proficiency Tests

QDOC/LAB/WI/ PT2

(Minus Section 11 Confirmation)

(A2LA Cert. No. 1914-01) Revised 04/15/2004

Page 1 of 1



SUMMARY: NAMES AND STRUCTURES OF ALL REPORTED COMPOUNDS

Laboratory code: 31

	atory co						
Sam. code	Cmpd. no*	Compound name	Chemical Abstract number	Compound Structure	Molecular formula	Schedule number	Comments**
O	1	Trichloronitromethane	76-06-2	CI CI—NO ₂	CCl ₃ NO ₂	3.A.04	
0	2	2-(N-Ethyl-N-propylamino)ethylchloride		cı	C ₇ H ₁₆ CIN	2.B.10	
O	3	2-(N,N-Diisopropylamino)ethylchloride	96-79-7	CI N	C ₈ H ₁₈ ClN	2.B.10	
O	4	2-(N,N-Diethylamino)ethylchloride	100-35-6	CI	C ₆ H ₁₄ CIN	2.B.10	
L	5	Triethanolamine	102-71-6	ОН	$C_6H_{15}NO_3$	3.B.17	
L	6	2-(N,N-Diisopropylamino)ethanol	96-80-0	но	C ₈ H ₁₉ NO	2.B.11	
S	7	Ethyldiethanolamine	139-87-7	ОН	C ₆ H ₁₅ NO ₂	3.B.15	
S	8	Methyldiethanolamine	105-59-9	ОН	C ₅ H ₁₃ NO ₂	3.B.16	
S	9	Triethanolamine	102-71-6	ОН	C ₆ H ₁₅ NO ₃	3.B.17	

^{*} Compound number defined by the participating laboratory and used throughout the report for the reported compound.

OPCW

Sample Prep, Part III, Page no. 65

** Explanation for the reporting of non-scheduled compounds, details can be added in the comment section of the report.

Note: There must be an unbroken chain of evidence linking each reported chemical to the original sample from which an aliquot was prepared and analyzed for the identification of this chemical.

SUMMARY: ANALYTICAL TECHNIQUES

Laboratory code: 31 Sample code(s): 0/31

Compound	Compound	Compound analysed	Analytical	Method	Method	Aliquot name
number*	name	as	technique	name	page no.	
1	Trichloronitromethane	original compound	GC/MS-EI	CW	70	CW-1-130-7-O
		methylated	GC/MS-CI	CW-CI	73	CW-1-130-7-O
		silylated				
		other:				
2	2-(N-Ethyl-N-propylamino)ethylchloride	original compound	GC/MS-EI	CW	76	CW-1-130-7-O
		methylated	GC/MS-CI	CW-CI	79	CW-1-130-7-O
		silylated				
		other:				
3	2-(N,N-Diisopropylamino)ethylchloride	original compound	GC/MS-EI	CW	82	CW-1-130-7-O
		methylated	GC/MS-CI	CW-CI	85	CW-1-130-7-O
		silylated				
		other:				
4	2-(N,N-Diethylamino)ethylchloride	original compound	GC/MS-EI	CW	88	CW-1-130-7-O
		methylated	GC/MS-CI	CW-CI	91	CW-1-130-7-O
		silylated				
		other:				

^{*} Compound number defined by the participating laboratory (see Summary: Names and Structures of All Reported Compounds);

SUMMARY: ANALYTICAL TECHNIQUES

Laboratory code: 31 Sample code(s): L/31

Compound	Compound	Compound analysed	Analytical	Method	Method	Aliquot name
number*	name	as	technique	name	page no.	
5	Triethanolamine	original compound	GC/MS-EI	TMS_A	96	CW-1-131-5-L
		methylated	GC/MS-CI	CW-CI-TM	99	CW-1-131-5-L
		⊠ silylated				
		other:				
6	2-(N,N-Diisopropylamino)ethanol	original compound	GC/MS-EI	TMS A	102	CW-1-131-4-L
		methylated	GC/MS-CI	CW-CI-TM	105	CW-1-131-4-L
		silylated				
		other:				
		original compound				
		methylated				
		silylated				
		other:				
		original compound				
		methylated				
		silylated				
		other:				

^{*} Compound number defined by the participating laboratory (see Summary: Names and Structures of All Reported Compounds);

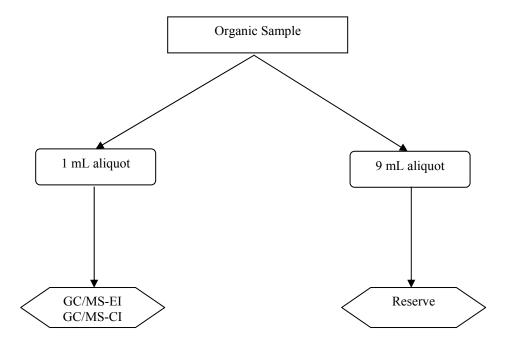
SUMMARY: ANALYTICAL TECHNIQUES

Laboratory code: 31 Sample code(s): S/31

Compound	Compound	Compound analysed	Analytical	Method	Method	Aliquot name
number*	name	as	technique	name	page no.	
7	Ethyldiethanolamine	original compound	GC/MS-EI	TMS_A	110	CW-1-131-2-S
		methylated	GC/MS-CI	CW-CI-TM	113	CW-1-131-2-S
		⊠ silylated				
		other:				
8	Methyldiethanolamine	original compound	GC/MS-EI	TMS A	116	CW-1-131-2-S
		methylated		CW-CI-TM	119	CW-1-131-2-S
		silylated			117	
		other:				
9	Triethanolamine	original compound	GC/MS-EI	TMS A	122	CW-1-131-2-S
		methylated 1	GC/MS-CI	_	125	CW-1-131-2-S
		silylated				
		other:				
		original compound				
		methylated				
		silylated				
		other:				

^{*} Compound number defined by the participating laboratory (see Summary: Names and Structures of All Reported Compounds);

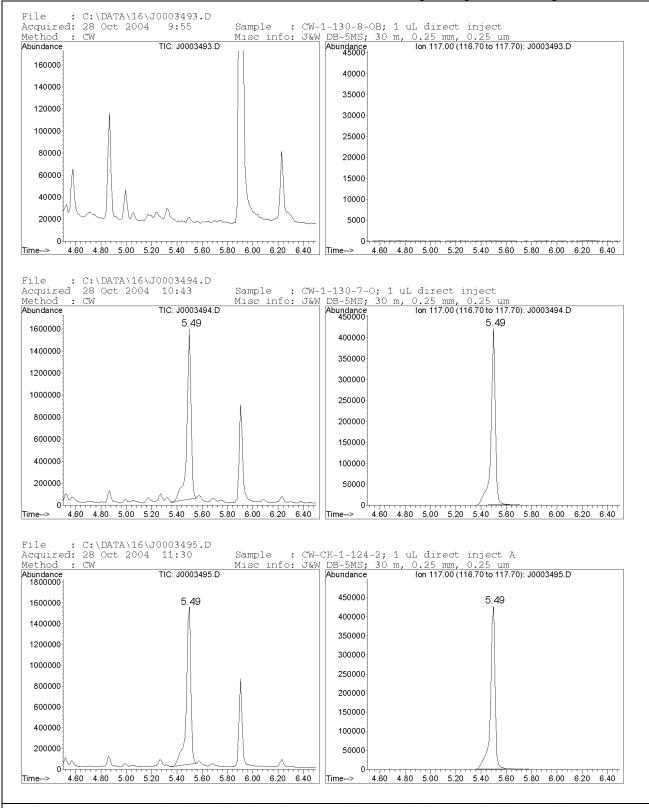
Laboratory code: 31 Sample code(s): 0/31 Sample Blank code: 0B/31									
1. Sample pr	eparation								
Sample/ Aliquot Code	Specification of Sample/ Type of Sample Preparation	Amount/ Volume	Sample Preparation Procedures	End Volume	Resulting Aliquot Code	Analytical Technique(s)			
O/31 OB/31	1 mL aliquot of each	1 mL	(none)	1 mL	CW-1-130-7-O CW-1-130-8-OB	GC/MS-EI			
2 Additional	information		1			<u> </u>			



Note: This flowchart is for visualization only; see the preceding sample preparation description page for sample aliquot numbers

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ampie co	oae(s): <u>O</u>	/31, OB/3	<u>1</u> Comp	ound number:	<u> </u>		
Aliquot codes:								
Sample: CW-1-130-7-O			Blank:	CW-1-130)-8-OB			
GC-EI-MS Method name:	CW							
GC-EI-WIS WIEthou hame.	CW							
METHOD DESCRIPTION								
Instrument Manufacturer	Agilent 6890/5973 GC/MSD							
and Type:								
Carrier gas:	⊠ He	\square N ₂	\square H ₂	Other:				
Flow rate:		ml/min		2 cm/s				
Flow control:	☐ Con	istant Pressu	ıre 🖂	Constant F	low			
Injection mode:			Split ratio					
	⊠ Spli	itless →	Splitless t	ime = 0.75 r	nin.			
Injector temperature:	250 °C							
Column phase:	5% dip	henyl 95% c	limethyl p	olysiloxane				
Column Length x ID x	30 m x	0.25 mm x 0).25 µm					
Film thickness:								
GC temperature	40 °C (3 min), 8 °C/min, 300 °C (3 min)							
programme:								
Solvent delay time:	3 min		Scan range:		30-600 m/z			
Electron energy:	70 eV		Scan tin		0.7 s			
Ionisation polarity:	⊠ Pos		Mass resolution:		0.7 u			
	∐ Neg	gative						
Comments:								
ANALYSIS								
Compound identified as:		☑ Original compound						
		☐ Methyl ester derivative						
	☐ TBDMS (t-Butyldimethylsilyl) derivative							
				ilyl) derivati	ve			
		Other d	erivative:					
Retention parameter used for	or	□ Retention	,	t)				
(peak) identification:		☐ Scan nu	ımber					
⊠ Compared to reference che	Source:	🛛 Own Sy	ynthesis	☐ Commercial				
☐ Compared to library spectr		Source:	OCAI	D (code:) NIST			
_			☐ Wiley	Ow	n Other:			
☐ Not compared to reference	Intense ions in spectrum are explained; interpretation is							
chemical or library spectru	m:		•	-	rmation derived from	m		
		closely	related che	emical(s):				
Comments:								



EI chromatograms supporting identification of compound 1; TIC on left; EIC (m/z 117) on right.

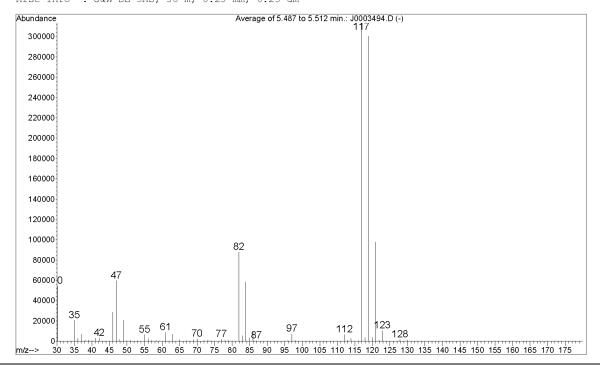
Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

Center: Chromatograms of Organic sample, aliquot CW-1-130-7-O from O/31, retention time 5.49 min.

Bottom: Chromatograms of authentic reference standard of **Trichloronitromethane**, retention time **5.49**

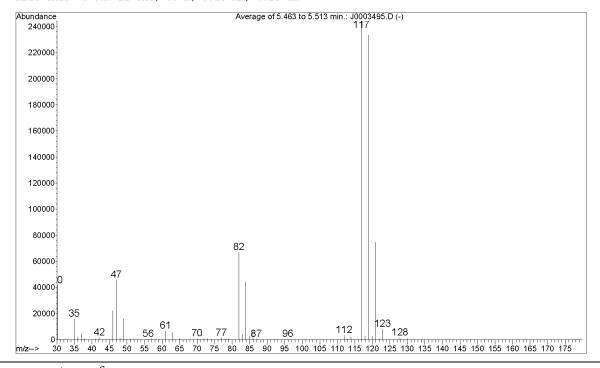
min.

File : C:\DATA\16\J0003494.D
Acquired : 28 Oct 2004 10:43 using AcqMe
Sample Name: CW-1-130-7-0; 1 uL direct inject
Misc Info : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um using AcqMethod CW



C:\DATA\16\J0003495.D

File : C:\DATA\16\J0003495.D Acquired : 28 Oct 2004 11:30 using AcqMetl Sample Name: CW-CK-1-124-2; 1 uL direct inject A using AcqMethod CW Misc Info : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um



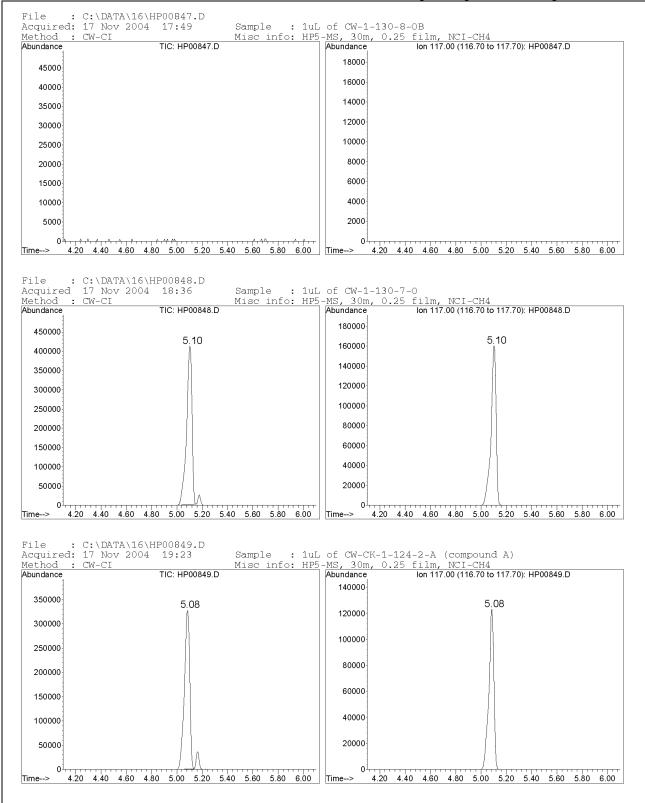
EI mass spectrum of:

Compound 1 in Organic sample O/31, aliquot CW-1-130-7-O Top:

Authentic reference standard of **Trichloronitromethane** corresponding to compound 1 (MW: Bottom: 163)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ample co	ode(s): O	/31, OB/3	<u>1</u> Comp	ound number: 1	
Aliquot codes:						
Sample: CW-1-130-7-O]	Blank:	CW-1-130	0-8-OB	
GC-CI-MS Method name:	CW-C	 [
METHOD DESCRIPTION						
Instrument Manufacturer	Agilent	6890/5973	GC/MSD			
and Type:						
Carrier gas:	⊠ He	\square N_2	\square H ₂	Other:		
Flow rate:		ml/min		2 cm/s		
Flow control:	☐ Con	istant Pressu	re 🖂	Constant F	low	
Injection mode:		it \rightarrow	Split ratio	=		
	⊠ Spli	itless →	Splitless ti	ime = 0.75 r	nin.	
Injector temperature:	250 °C					
Column phase:	5% dip	henyl 95% d	limethyl po	olysiloxane		
Column Length x ID x	30 m x	0.25 mm x 0).25 μm			
Film thickness:						
GC temperature	40 °C (3 min), 8 °C	/min, 300	°C (3 min)		
programme:						
Reaction gas:	⊠ Met	hane 🗌 Is	sobutane	Ammo:	nia 🗌 Other:	
Solvent delay time:	3 min		Scan rar	ige:	50-550 m/z	
Electron energy:	235 eV		Scan tim	ie:	0.35 s	
Ionisation polarity:	☐ Pos		Mass res	solution:	0.7 u	
	Neg	gative				
Comments:						
ANALYSIS						
Compound identified as:		☐ Original				
			ester deriv			
			` -		l) derivative	
				lyl) derivati	ve	
			erivative:			
Retention parameter used for	or	Retention	*	t)		
(peak) identification:		☐ Scan nu				
☐ Compared to reference che	mical:	Source:	⊠ Own Sy	nthesis	☐ Commercial	
☐ Compared to library spectr	um:	Source:		O (code:) NIST	
			☐ Wiley			
Not compared to reference			_	_	ained; interpretation is	
chemical or library spectru	m:		•		rmation derived from	
		closely 1	related che	emical(s):		
Comments:		1				



CI chromatograms supporting identification of compound 1; TIC on left; EIC (m/z 117) on right.

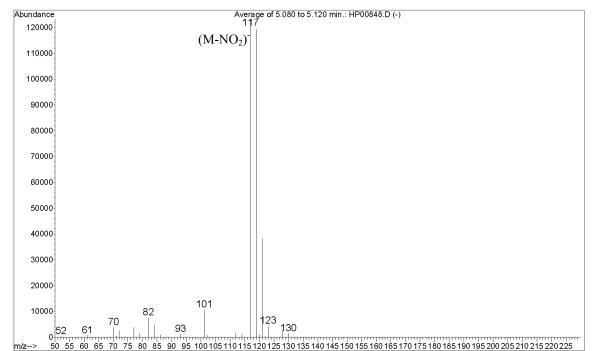
Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

Center: Chromatograms of Organic sample, aliquot CW-1-130-7-O from O/31, retention time 5.10 min.

Bottom: Chromatograms of authentic reference standard of **Trichloronitromethane**, retention time **5.08**

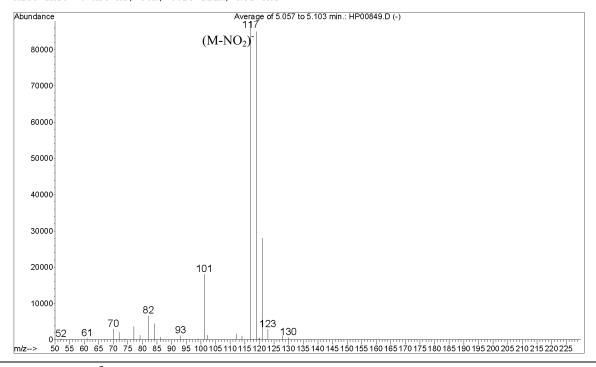
min.

File : C:\DATA\16\HP00848.D
Acquired : 17 Nov 2004 18:36 using Acc
Sample Name: 1uL of CW-1-130-7-0
Misc Info : HP5-MS, 30m, 0.25 film, NCI-CH4 using AcqMethod CW-CI



File : C:\DATA\16\HP00849.D Acquired : 17 Nov 2004 19:23 using AcqMet Sample Name: 1uL of CW-CK-1-124-2-A (compound A) Acquired using AcqMethod CW-CI

Misc Info : HP5-MS, 30m, 0.25 film, NCI-CH4



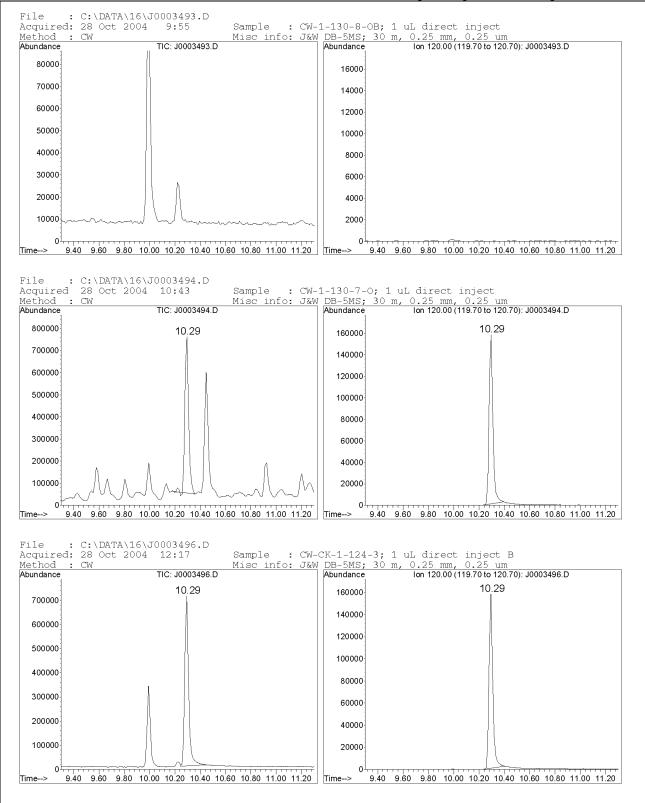
CI mass spectrum of:

Compound 1 in Organic sample O/31, aliquot CW-1-130-7-O Top:

Authentic reference standard of **Trichloronitromethane** corresponding to compound 1 (MW: Bottom: 163)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ampie co	oae(s): <u>O</u>	/31, OB/3	<u>1</u> Comp	ound number: <u>2</u>			
Aliquot codes:								
Sample: CW-1-130-7-O			Blank:	CW-1-130)-8-OB			
GC-EI-MS Method name:	CW							
GC-E1-WIS WIEthou hame.	CW							
METHOD DESCRIPTION								
Instrument Manufacturer	Agilent 6890/5973 GC/MSD							
and Type:								
Carrier gas:	⊠ He	\square N ₂	\square H ₂	Other:				
Flow rate:		ml/min		2 cm/s				
Flow control:	☐ Con	istant Pressu	re 🖂	Constant F	low			
Injection mode:	☐ Spli	it \rightarrow	Split ratio	=				
	⊠ Spli	tless →	Splitless t	ime = 0.75 r	nin.			
Injector temperature:	250 °C							
Column phase:	5% dip	henyl 95% d	limethyl p	olysiloxane				
Column Length x ID x	30 m x	0.25 mm x 0).25 µm					
Film thickness:								
GC temperature	40 °C (3 min), 8 °C	7/min, 300	°C (3 min)				
programme:								
Solvent delay time:	3 min		Scan range:		30-600 m/z			
Electron energy:	70 eV		Scan tin		0.7 s			
Ionisation polarity:	⊠ Pos	itive	Mass resolution:		0.7 u			
	☐ Neg	gative						
Comments:								
ANALYSIS								
Compound identified as:		☑ Original compound						
		☐ Methyl ester derivative						
	☐ TBDMS (t-Butyldimethylsilyl) derivative							
				ilyl) derivati	ve			
		Other d	erivative:					
Retention parameter used for	or	□ Retention	,	t)				
(peak) identification:								
⊠ Compared to reference che	Source:	⊠ Own Sy	ynthesis	☐ Commercial				
☐ Compared to library spectr	Source:	OCAI	O (code:) NIST				
_	☐ Wiley ☐ Own ☐ Other:							
☐ Not compared to reference		Intense ions in spectrum are explained; interpretation is						
chemical or library spectru	m:		•	-	rmation derived from			
		closely	related che	emical(s):				
Comments:								



EI chromatograms supporting identification of compound 2; TIC on left; EIC (m/z 120) on right.

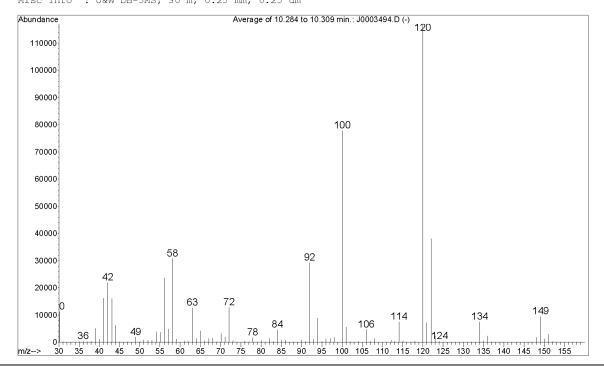
Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

Center: Chromatograms of Organic sample, aliquot CW-1-130-7-O from O/31, retention time 10.29 min.

Bottom: Chromatograms of authentic reference standard of 2-(N-Ethyl-N-propylamino)ethylchloride,

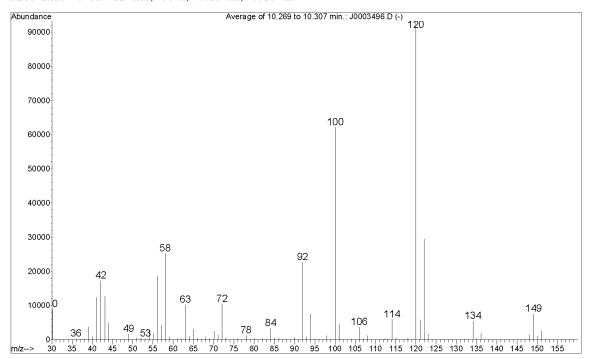
retention time 10.29 min.

File : C:\DATA\16\J0003494.D
Acquired : 28 Oct 2004 10:43 using AcqMethod CW
Sample Name: CW-1-130-7-0; 1 uL direct inject
Misc Info : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um



File : C:\DATA\16\J0003496.D

Acquired: 28 Oct 2004 12:17 using AcqMethod CW Sample Name: CW-CK-1-124-3; 1 uL direct inject B Misc Info: J&W DB-5MS; 30 m, 0.25 mm, 0.25 um



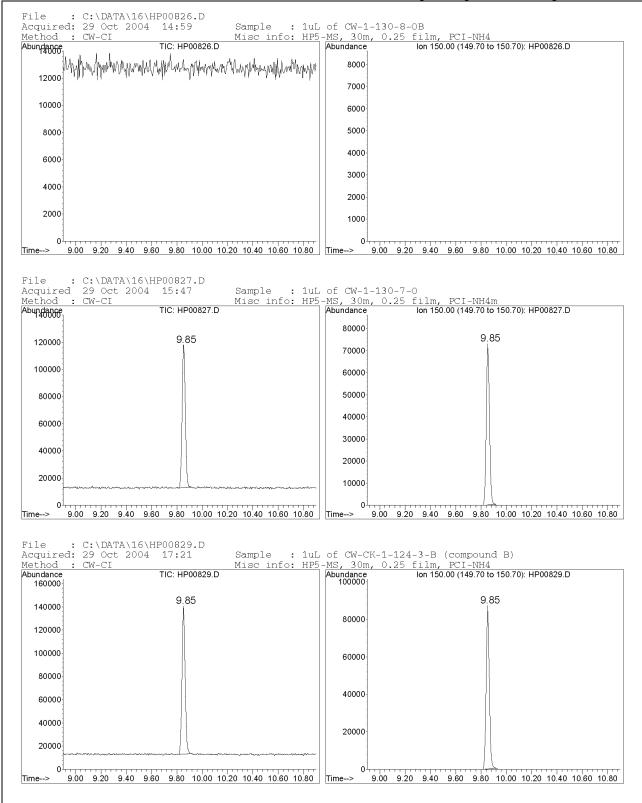
EI mass spectrum of:

Top: Compound 2 in Organic sample O/31, aliquot CW-1-130-7-O

Bottom: Authentic reference standard of **2-(N-Ethyl-N-propylamino)ethylchloride** corresponding to compound **2** (MW: **149**)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ample co	ode(s): O	/31, OB/3	<u>1</u> Comp	ound number: <u>2</u>	
Aliquot codes:						
Sample: CW-1-130-7-O			Blank:	CW-1-130	0-8-OB	
GC-CI-MS Method name:	CW-C	 [
METHOD DESCRIPTION						
Instrument Manufacturer	Agilent	t 6890/5973	GC/MSD			
and Type:	ì					
Carrier gas:	⊠ He	\square N_2	\square H ₂	Other:		
Flow rate:		ml/min	\boxtimes 32	2 cm/s		
Flow control:	☐ Con	istant Pressu	re 🖂	Constant F	low	
Injection mode:		it \rightarrow	Split ratio	=		
	⊠ Spli	itless →	Splitless t	ime = 0.75 r	nin.	
Injector temperature:	250 °C					
Column phase:	5% dip	henyl 95% d	limethyl p	olysiloxane		
Column Length x ID x	30 m x	0.25 mm x 0).25 μm			
Film thickness:						
GC temperature	40 °C (3 min), 8 °C	/min, 300	°C (3 min)		
programme:						
Reaction gas:	☐ Met	hane 🗌 Is	sobutane	⊠ Ammo	nia 🗌 Other:	
Solvent delay time:	3 min		Scan rai	nge:	50-550 m/z	
Electron energy:	235 eV	·	Scan tin	ne:	0.35 s	
Ionisation polarity:	⊠ Pos		Mass res	solution:	0.7 u	
	☐ Neg	gative				
Comments:						
ANALYSIS						
Compound identified as:		☐ Original				
			ester deriv			
			` -	dimethylsily	/	
				ilyl) derivati	ve	
			erivative:			
Retention parameter used for	or	Retention	,	t)		
(peak) identification:		☐ Scan nu				
□ Compared to reference che	mical:		⊠ Own Sy	<u></u>	☐ Commercial	
☐ Compared to library spectr	um:	Source:		O (code:) NIST	
			☐ Wiley			
Not compared to reference			_	_	ained; interpretation is	
chemical or library spectru	m:		•		rmation derived from	
Comments:		closely 1	related che	emical(s):		
L CMMente.		1				



CI chromatograms supporting identification of compound 2; TIC on left; EIC (m/z 150) on right.

Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

Center: Chromatograms of Organic sample, aliquot CW-1-130-7-O from O/31, retention time 9.85 min.

Bottom: Chromatograms of authentic reference standard of 2-(N-Ethyl-N-propylamino)ethylchloride,

retention time 9.85 min.

File : C:\DATA\16\HP00827.D
Acquired : 29 Oct 2004 15:47 using Acql
Sample Name: 1uL of CW-1-130-7-0
Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH4m using AcqMethod CW-CI

Average of 9.840 to 9.869 min.: HP00827.D (-) 150 Abundance 50000 $(M+H)^+$ 45000 40000 35000 30000 25000 20000 15000 10000

114

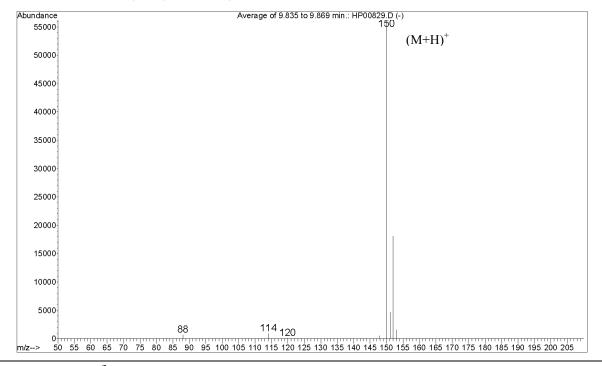
50 55 60 65 70 75 80 85 90 95 100 105 110 115 120 125 130 135 140 145 150 155 160 165 170 175 180 185 190 195 200 205

5000

using AcqMethod CW-CI Acquired

88

File : C:\DATA\16\HP00829.D
Acquired : 29 Oct 2004 17:21 using AcqMet
Sample Name: 1uL of CW-CK-1-124-3-B (compound B) Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH4



CI mass spectrum of:

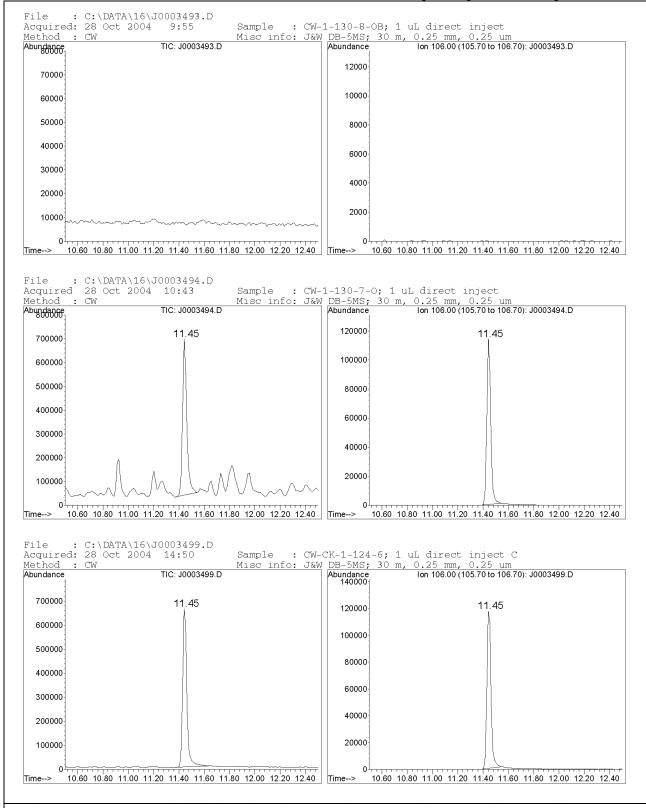
Compound 2 in Organic sample O/31, aliquot CW-1-130-7-O Top:

Bottom: Authentic reference standard of 2-(N-Ethyl-N-propylamino)ethylchloride corresponding to

compound 2 (MW: 149)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ample code(s):	<u>O/31, OB/31</u> Com	ipound number: 3					
Aliquot codes:								
Sample: CW-1-130-7-O		Blank: CW-1-1	30-8-OB					
GC-EI-MS Method name: CW								
METHOD DESCRIPTION								
Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD							
Carrier gas:	\square He \square N ₂	☐ H ₂ ☐ Other	·:					
Flow rate:	□ ml/min	≥ 32 cm/s						
Flow control:	☐ Constant Press	sure 🛛 Constant	Flow					
Injection mode:	\square Split \rightarrow	Split ratio =						
	\boxtimes Splitless \rightarrow	Splitless time = 0.75	i min.					
Injector temperature:	250 °C							
Column phase:	5% diphenyl 95%	dimethyl polysiloxan	e					
Column Length x ID x	30 m x 0.25 mm x							
Film thickness:	· ·							
GC temperature	40 °C (3 min), 8 °	C/min, 300 °C (3 min)					
programme:								
Solvent delay time:	3 min	Scan range:	30-600 m/z					
Electron energy:	70 eV	Scan time:	0.7 s					
Ionisation polarity:	☑ Positive☑ Negative	Mass resolution:	0.7 u					
Comments:								
ANALYSIS								
Compound identified as:	☐ Methy ☐ TBDM ☐ TMS (☑ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative: 						
Retention parameter used for Retention time (Rt)								
(peak) identification:		umber						
⊠ Compared to reference che		Own Synthesis						
☐ Compared to library spectr	rum: Source:	,	☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:					
☐ Not compared to reference chemical or library spectru	m: suppor		plained; interpretation is formation derived from					
Comments:								



EI chromatograms supporting identification of compound 3; TIC on left; EIC (m/z 106) on right.

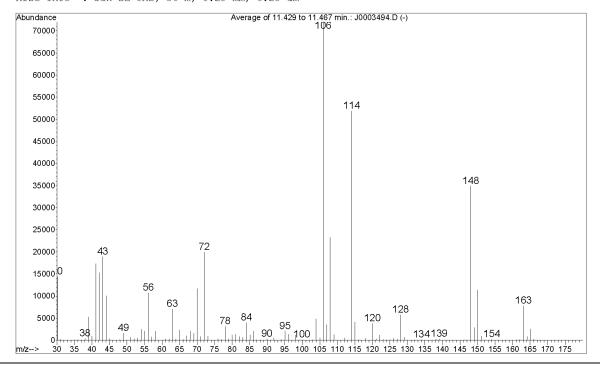
Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

Center: Chromatograms of Organic sample, aliquot CW-1-130-7-O from O/31, retention time 11.45 min.

Bottom: Chromatograms of authentic reference standard of **2-(N,N-Diisopropylamino)ethylchloride**,

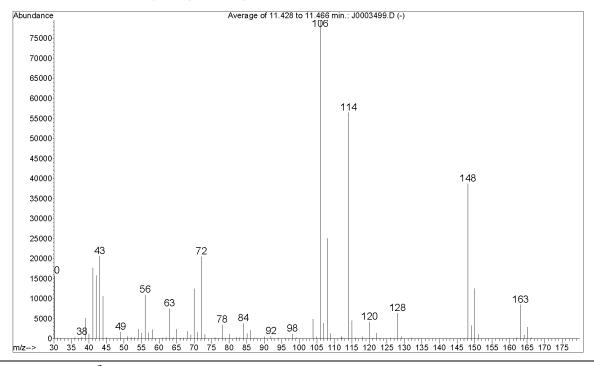
retention time 11.45 min.

File : C:\DATA\16\J0003494.D
Acquired : 28 Oct 2004 10:43 using AcqMe
Sample Name: CW-1-130-7-0; 1 uL direct inject
Misc Info : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um using AcqMethod CW



C:\DATA\16\J0003499.D

File : C:\DATA\16\J0003499.D Acquired : 28 Oct 2004 14:50 using AcqMetl Sample Name: CW-CK-1-124-6; 1 uL direct inject C using AcqMethod CW Misc Info : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um



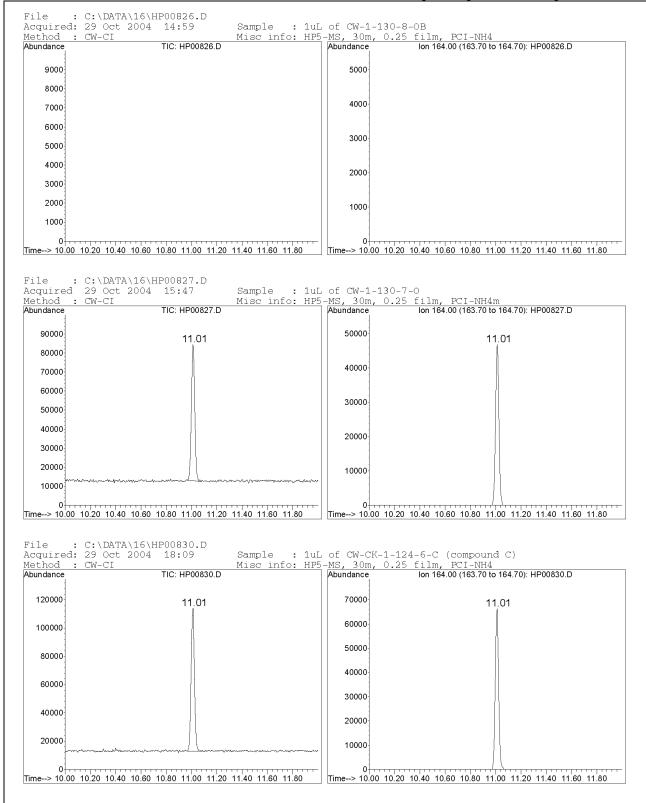
EI mass spectrum of:

Compound 3 in Organic sample O/31, aliquot CW-1-130-7-O Top:

Authentic reference standard of 2-(N,N-Diisopropylamino)ethylchloride corresponding to Bottom: compound 3 (MW: 163)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ample co	ode(s): O	/31, OB/3	<u>1</u> Comp	ound number: 3	
Aliquot codes:						
Sample: CW-1-130-7-O		,	Blank:	CW-1-130	0-8-OB	
GC-CI-MS Method name:	CW-C					
METHOD DESCRIPTION						
Instrument Manufacturer	Agilent	6890/5973	GC/MSD			
and Type:	ì					
Carrier gas:	⊠ He	\square N_2	\square H ₂	Other:		
Flow rate:		ml/min	⊠ 32	2 cm/s		
Flow control:	☐ Con	istant Pressu	re 🖂	Constant F	low	
Injection mode:		$it \rightarrow$	Split ratio	=		
	⊠ Spli	itless →	Splitless t	ime = 0.75 r	nin.	
Injector temperature:	250 °C					
Column phase:	5% dip	henyl 95% d	limethyl p	olysiloxane		
Column Length x ID x	30 m x	0.25 mm x 0).25 μm			
Film thickness:						
GC temperature	40 °C (3 min), 8 °C	/min, 300	°C (3 min)		
programme:						
Reaction gas:	☐ Met	hane 🗌 Is	sobutane	⊠ Ammo	nia 🗌 Other:	
Solvent delay time:	3 min		Scan ran	nge:	50-550 m/z	
Electron energy:	235 eV		Scan tim	ne:	0.35 s	
Ionisation polarity:	⊠ Pos		Mass res	solution:	0.7 u	
	☐ Neg	gative				
Comments:						
ANALYSIS						
Compound identified as:		□ Original				
			ester deriv			
			` -	dimethylsily	/	
				ilyl) derivati	ve	
			erivative:			
Retention parameter used for	or	Retention	,	t)		
(peak) identification:		☐ Scan nu	mber			
☐ Compared to reference che	mical:	Source:	Own Sy	ynthesis		
☐ Compared to library spectr	um:	Source:		O (code:) NIST	
			☐ Wiley			
Not compared to reference			_	_	ained; interpretation is	
chemical or library spectru	m:		•		rmation derived from	
		closely 1	related che	emical(s):		
Comments:						



CI chromatograms supporting identification of compound 3; TIC on left; EIC (m/z 164) on right.

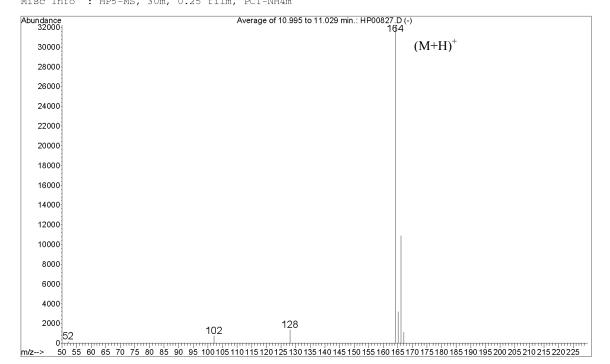
Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

Center: Chromatograms of Organic sample, aliquot CW-1-130-7-O from O/31, retention time 11.01 min.

Bottom: Chromatograms of authentic reference standard of **2-(N,N-Diisopropylamino)ethylchloride**,

retention time 11.01 min.

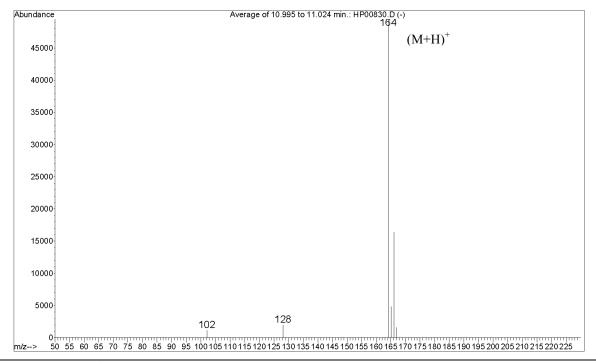
File : C:\DATA\16\HP00827.D
Acquired : 29 Oct 2004 15:47 using AcqMethod CW-CI
Sample Name: 1uL of CW-1-130-7-0
Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH4m



File : C:\DATA\16\HP00830.D

Acquired: 29 Oct 2004 18:09 using AcqMethod CW-CI Sample Name: 1uL of CW-CK-1-124-6-C (compound C)

Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH4



CI mass spectrum of:

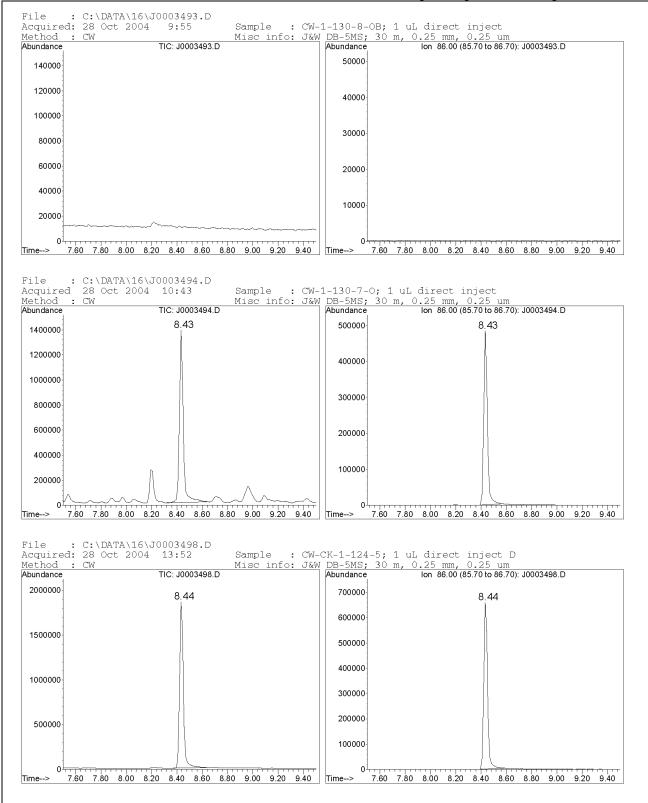
Top: Compound 3 in Organic sample O/31, aliquot CW-1-130-7-O

Bottom: Authentic reference standard of 2-(N,N-Diisopropylamino)ethylchloride corresponding to

compound **3** (MW: **163**)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ampie co	oae(s): <u>O</u>	9/31, OB/3	<u>1</u> Comp	ound number: <u>2</u>	<u>4</u> _		
Aliquot codes:								
Sample: CW-1-130-7-O			Blank:	CW-1-130)-8-OB			
GC-EI-MS Method name:	CW							
GC-E1-MS Method hame:	CW							
METHOD DESCRIPTION								
Instrument Manufacturer	Agilent 6890/5973 GC/MSD							
and Type:								
Carrier gas:	⊠ He	\square N ₂	\square H ₂	Other:				
Flow rate:		ml/min		2 cm/s				
Flow control:	☐ Con	istant Pressu	ıre 🖂	Constant F	low			
Injection mode:			Split ratio					
	⊠ Spli	itless →	Splitless t	ime = 0.75 r	nin.			
Injector temperature:	250 °C							
Column phase:	5% dip	henyl 95% c	dimethyl p	olysiloxane				
Column Length x ID x	30 m x	0.25 mm x 0).25 µm					
Film thickness:								
GC temperature	40 °C (3 min), 8 °C/min, 300 °C (3 min)							
programme:								
Solvent delay time:	3 min		Scan range:		30-600 m/z			
Electron energy:	70 eV		Scan tim		0.7 s			
Ionisation polarity:	⊠ Pos		Mass resolution:		0.7 u			
	∐ Neg	gative						
Comments:								
ANALYSIS								
Compound identified as:		⊠ Original compound						
		☐ Methyl ester derivative						
	☐ TBDMS (t-Butyldimethylsilyl) derivative							
				lyl) derivati	ve			
		☐ Other d	erivative:					
l =	Retention parameter used for ⊠ Retention time (Rt)							
(peak) identification:								
⊠ Compared to reference che	Source:	Own Sy	nthesis					
☐ Compared to library spectr	Source:	OCAI	O (code:) NIST				
_	☐ Wiley ☐ Own ☐ Other:							
☐ Not compared to reference		Intense ions in spectrum are explained; interpretation is						
chemical or library spectru	m:		-	-	rmation derived from	1		
		closely	related che	emical(s):				
Comments:								



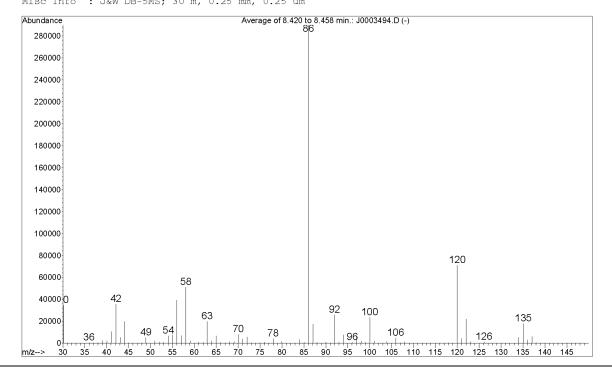
EI chromatograms supporting identification of compound 4; TIC on left; EIC (m/z 86) on right.

Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

Center: Chromatograms of Organic sample, aliquot CW-1-130-7-O from O/31, retention time 8.43 min.

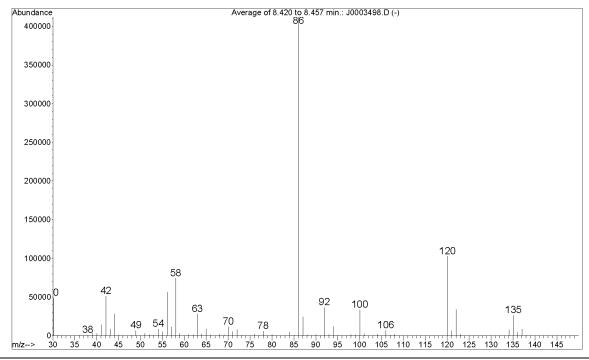
Bottom: Chromatograms of authentic reference standard of **2-(N,N-Diethylamino)ethylchloride**, retention time **8.44** min.

File : C:\DATA\16\J0003494.D
Acquired : 28 Oct 2004 10:43 using AcqMe
Sample Name: CW-1-130-7-0; 1 uL direct inject
Misc Info : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um using AcqMethod CW



C:\DATA\16\J0003498.D

Acquired : 28 Oct 2004 13:52 using AcqMetl Sample Name: CW-CK-1-124-5; 1 uL direct inject D using AcqMethod CW Misc Info : J&W DB-5MS; 30 m, 0.25 mm, 0.25 um



EI mass spectrum of:

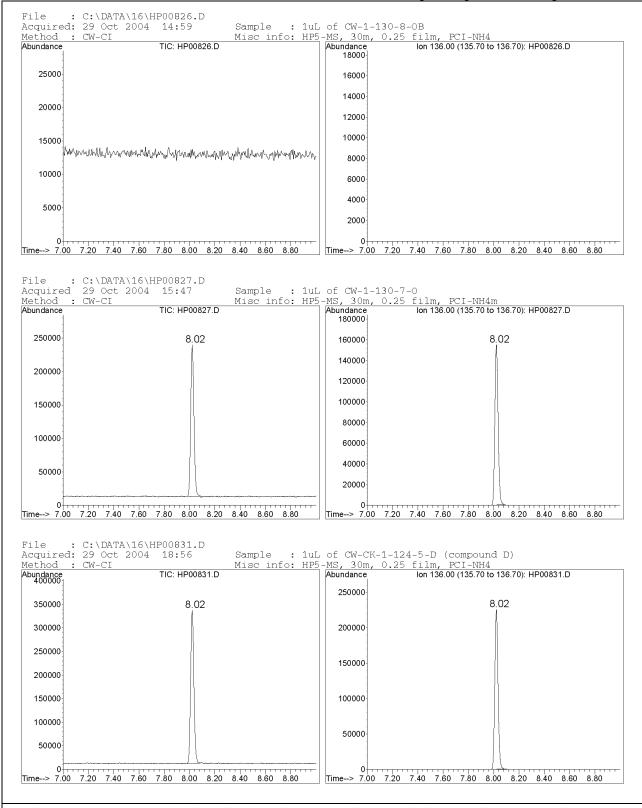
Compound 4 in Organic sample O/31, aliquot CW-1-130-7-O Top:

Bottom: Authentic reference standard of 2-(N,N-Diethylamino)ethylchloride corresponding to

compound 4 (MW: 135)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ample co	ode(s): O	/31, OB/3	<u>1</u> Comp	ound number: 4	
Aliquot codes:						
Sample: CW-1-130-7-O		,	Blank:	CW-1-130	0-8-OB	
GC-CI-MS Method name:	CW-C	T				
METHOD DESCRIPTION	0 11 0					
Instrument Manufacturer	Agilent	t 6890/5973	GC/MSD			
and Type:	1 1811411	. 000 0, 00 , 10	00/1/122			
Carrier gas:	⊠ He	\square N_2	\square H ₂	Other:		
Flow rate:		ml/min	⊠ 32	2 cm/s		
Flow control:	☐ Cor	stant Pressu	ire 🖂	Constant F	low	
Injection mode:	☐ Spli	it \rightarrow	Split ratio	=		
	⊠ Spli	itless →	Splitless t	ime = 0.75 r	nin.	
Injector temperature:	250 °C					
Column phase:	5% dip	henyl 95% d	limethyl p	olysiloxane		
Column Length x ID x	30 m x	0.25 mm x 0).25 µm			
Film thickness:						
GC temperature	40 °C (3 min), 8 °C/min, 300 °C (3 min)					
programme:						
Reaction gas:		hane 🗌 Is	sobutane	⊠ Ammo		
Solvent delay time:	3 min		Scan rai		50-550 m/z	
Electron energy:	235 eV		Scan tin		0.35 s	
Ionisation polarity:	⊠ Pos		Mass res	solution:	0.7 u	
	∐ Neg	gative				
Comments:						
ANALYSIS		·				
Compound identified as:		⊠ Original				
			ester deriv		N 1 ' ' '	
			` -	dimethylsily	/	
			rimetnyisi erivative:	ilyl) derivati	ve	
D-44:				4)		
Retention parameter used for (peak) identification:	or	□ Retention □ Scan nu	*	τ)		
☐ Compared to reference che	mical:	Source:	Own Sy	ynthesis	⊠ Commercial	
Compared to library spectr		Source:		O (code:) □ NIST	
	Wiley Own Other:					
Not compared to reference		Intense ion	s in spectr	um are expl	ained; interpretation is	
chemical or library spectru	m:		•		rmation derived from	
		closely 1	related che	emical(s):		
Comments:						



CI chromatograms supporting identification of compound 4; TIC on left; EIC (m/z 136) on right.

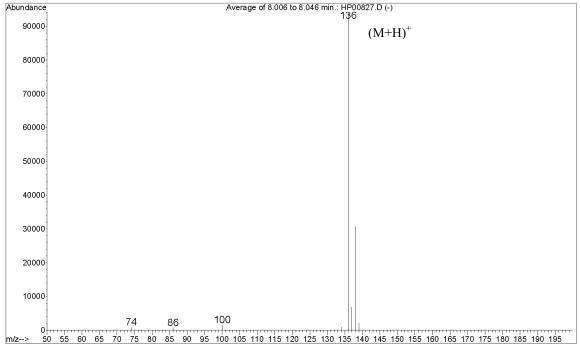
Top: Chromatograms of Organic blank, aliquot **CW-1-130-8-OB** from **OB/31**.

Center: Chromatograms of Organic sample, aliquot CW-1-130-7-O from O/31, retention time 8.02 min.

Bottom: Chromatograms of authentic reference standard of **2-(N,N-Diethylamino)ethylchloride**, retention time **8.02** min.

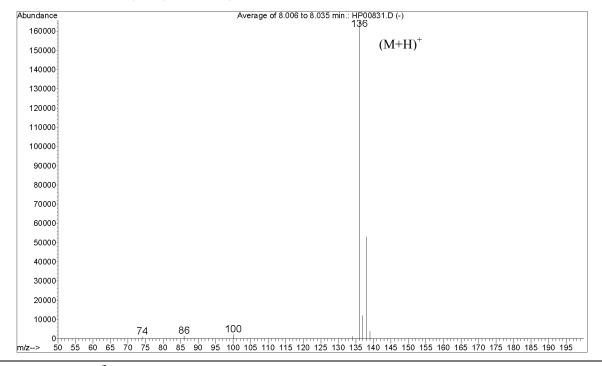
File : C:\DATA\16\HP00827.D
Acquired : 29 Oct 2004 15:47 using Acquired Sample Name: 1uL of CW-1-130-7-0
Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH4m using AcqMethod CW-CI

Abundance 90000



File : C:\DATA\16\HP00831.D Acquired : 29 Oct 2004 18:56 using AcqMet Sample Name: 1uL of CW-CK-1-124-5-D (compound D) using AcqMethod CW-CI Acquired

Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH4



CI mass spectrum of:

Compound 4 in Organic sample O/31, aliquot CW-1-130-7-O Top:

Bottom: Authentic reference standard of 2-(N,N-Diethylamino)ethylchloride corresponding to

compound 4 (MW: 135)

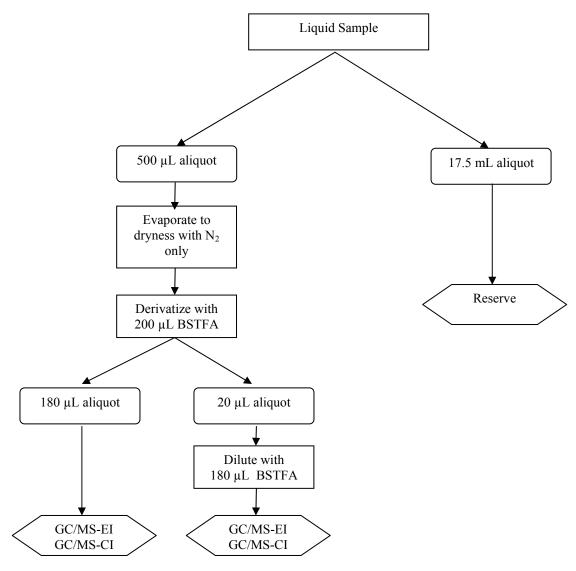
OPCW	Sample Prep, Part III, Page no. 94							
SAMPLE PREPARATION DESCRIPTION								
Laboratory code: 31 Sample code(s): I	/31 Sample Blank code: LB/31							

1. Sample preparation

Sample/ Aliquot Code	Specification of Sample/ Type of Sample Preparation	Amount/ Volume	Sample Preparation Procedures	End Volume	Resulting Aliquot Code	Analytical Technique(s)
L/31 LB/31	TMS derivative of sample	500 μL	500 μL evaporated to dryness (N ₂ , no heat). Added 500 μL BSTFA. Reacted at 60°C for 30 minutes.	500 μL	CW-1-131-4-L CW-1-131-3-LB	GC/MS-EI GC/MS-CI
CW-1-131-4-L	Dilution	20 μL	Diluted TMS derivitized samples with 180 µL BSTFA.	200 μL	CW-1-131-5-L	GC/MS-EI GC/MS-CI

2. Additional information

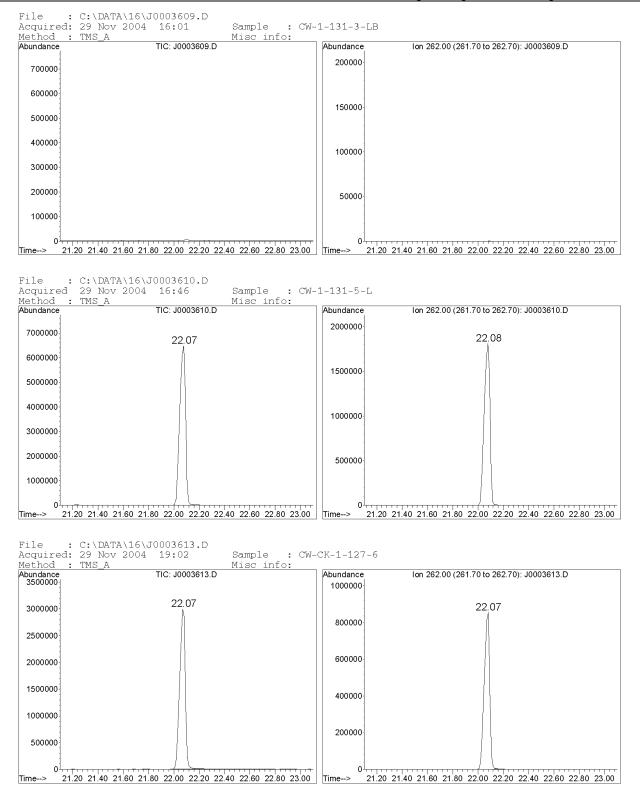
Note: sample CW-1-131-4-L was diluted with the same batch of BSTFA with which it had been derivitized to produce sample CW-1-131-5-L. Blank CW-1-131-3-LB was used directly as a blank (worst case – any scheduled compounds would be seen 10x as compared to the diluted sample).



Note: This flowchart is for visualization only; see the preceding sample preparation description page for sample aliquot numbers

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ample code(s	s): <u>L</u>	/31, LB/31	Comp	ound number:	<u>5</u>		
Aliquot codes:								
Sample: CW-1-131-5-L		-	Blank:	CW-1-131	1-3-LB			
GC-EI-MS Method name: TMS_A								
METHOD DESCRIPTION								
Instrument Manufacturer and Type:	ufacturer Agilent 6890/5973 GC/MSD							
Carrier gas:	⊠He	\square He \square N ₂ \square H ₂ \square Other:						
Flow rate:		nl/min		3 cm/s				
Flow control:		t Pressu		Constant F	low			
Injection mode:	☐ Split		Split ratio					
J			-	ime = 0.70	min.			
Injector temperature:	250 °C							
Column phase:	5% dipheny	ıl 95% d	limethyl po	olysiloxane				
Column Length x ID x	30 m x 0.25	mm x 0).25 μm					
Film thickness:								
GC temperature	70 °C (8 mi	n), 8 °C	7/min, 300	°C (3 min)				
programme:								
Solvent delay time:	8 min		Scan rar		40-600 m/z			
Electron energy:	70 eV		Scan tim		0.7 s			
Ionisation polarity:	☑ Positive☑ Negative		Mass res	solution:	0.7 u			
Comments:								
ANALYSIS								
Compound identified as:		Original	l compoun	d				
		☐ Methyl ester derivative						
		☐ TBDMS (t-Butyldimethylsilyl) derivative						
		☐ TMS (Trimethylsilyl) derivative						
	☐ Other derivative:							
Retention parameter used for ⊠ Retention time (Rt)								
(peak) identification: Scan number								
⊠ Compared to reference che	mical: Sou	arce :	Own Sy	nthesis				
☐ Compared to library spectr		arce :	☐ OCAI	O (code:) 🗌 NIST			
			☐ Wiley	Ow	vn Other:			
☐ Not compared to reference		Intense ions in spectrum are explained; interpretation is						
chemical or library spectru	m: s	supporte	ed by the s	pectral info	rmation derived from			
	(closely 1	related che	emical(s):				
Comments:								



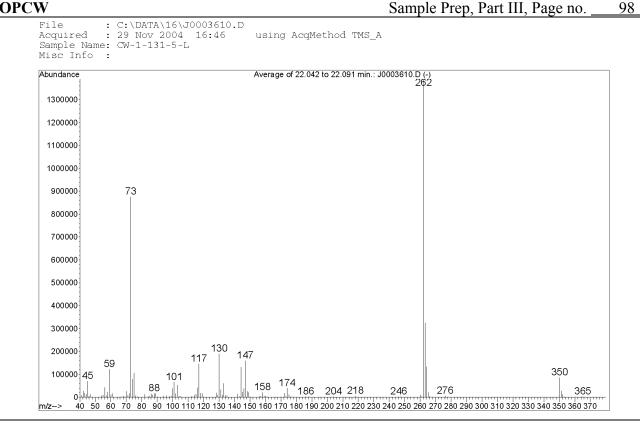
EI chromatograms supporting identification of compound 5; TIC on left; EIC (m/z 262) on right.

Top: Chromatograms of Liquid blank, aliquot **CW-1-131-3-LB** from **LB/31**.

Center: Chromatograms of Liquid sample, aliquot CW-1-131-5-L from L/31, retention time 22.07 min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Triethanolamine**

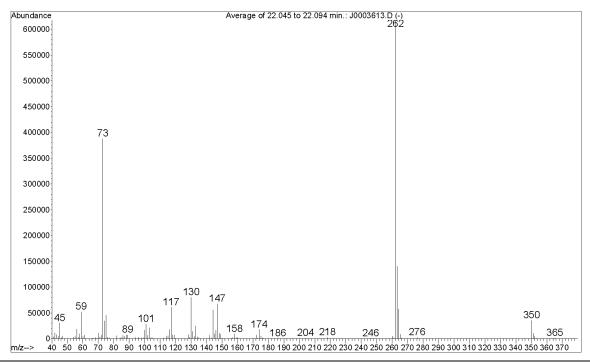
[tris(2-trimethylsiloxyethyl)amine], retention time **22.07** min.



File : C:\DATA\16\J0003613.D Acquired : 29 Nov 2004 19:02 Sample Name: CW-CK-1-127-6

using AcqMethod TMS_A

Misc Info



EI mass spectrum of:

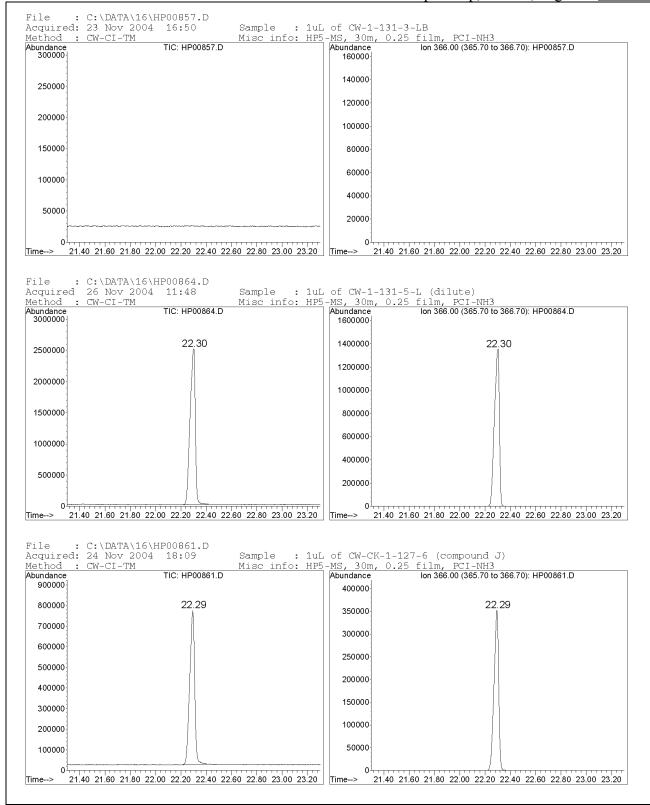
Compound 5 in Liquid sample L/31, aliquot CW-1-131-5-L Top:

Bottom: TMS derivative of the authentic reference standard of **Triethanolamine** trimethylsiloxyethyl)amine] corresponding to compound 5 (MW: 365)

[tris(2-

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ample co	ode(s): <u>L/</u>	/31, LB/31	<u> </u>	ound number: <u>5</u>	
Aliquot codes:						
Sample: CW-1-131-5-L			Blank:	CW-1-131	-3-LB	
GC-CI-MS Method name:	CW-C	I-TM				
METHOD DESCRIPTION						
Instrument Manufacturer	Agilent	t 6890/5973	GC/MSD			
and Type:	J					
Carrier gas:	⊠ He	\square N ₂	\square H ₂	Other:		
Flow rate:		ml/min		2 cm/s		
Flow control:	☐ Con	istant Pressu	re 🖂	Constant F	low	
Injection mode:	☐ Spli	it \rightarrow	Split ratio	=		
			Splitless t	ime = 0.75 r	nin.	
Injector temperature:	250 °C					
Column phase:	5% dip	henyl 95% d	limethyl p	olysiloxane		
Column Length x ID x	30 m x	0.25 mm x 0).25 μm			
Film thickness:						
GC temperature	70 °C (8 min), 8 °C/min, 300 °C (3 min)					
programme:						
Reaction gas:		hane 🗌 Is	sobutane	⊠ Ammo		
Solvent delay time:	8 min		Scan rai		50-550 m/z	
Electron energy:	235 eV		Scan tin		0.35 s	
Ionisation polarity:	⊠ Pos		Mass res	solution:	0.7 u	
C	∐ Neg	gative				
Comments:						
ANALYSIS		<u> </u>				
Compound identified as:		_	l compoun			
		☐ Methyl ester derivative				
				dimethylsily		
				ilyl) derivati	ve	
D. C. L.			erivative:	4)		
Retention parameter used for (peak) identification:	Retention time (Rt) Scan number					
Compared to reference che	mical•	Source :	Own Sy	unthesis	⊠ Commercial	
Compared to library spectr		Source:		O (code:) NIST	
sompared to notary speed	W1111•	200100.	☐ Wiley	`	/ -	
Not compared to reference)	Intense ion			ained; interpretation is	
chemical or library spectru			_	_	rmation derived from	
			related che			
Comments:				` (



CI chromatograms supporting identification of compound 5; TIC on left; EIC (m/z 366) on right.

Chromatograms of Liquid blank, aliquot CW-1-131-3-LB from LB/31. Top:

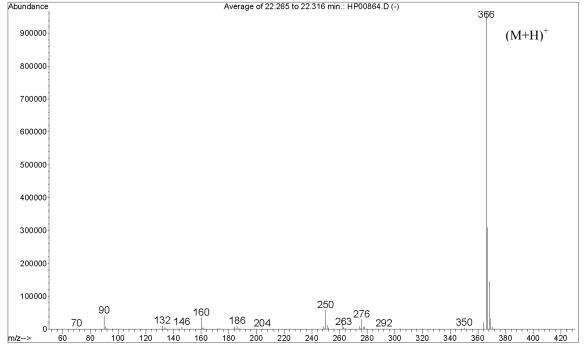
Center: Chromatograms of Liquid sample, aliquot CW-1-131-5-L from L/31, retention time 22.30 min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Triethanolamine**

[tris(2-trimethylsiloxyethyl)amine], retention time 22.29 min.

File : C:\DATA\16\HP00864.D
Acquired : 26 Nov 2004 11:48 using Ac
Sample Name: 1uL of CW-1-131-5-L (dilute)
Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH3 using AcqMethod CW-CI-TM

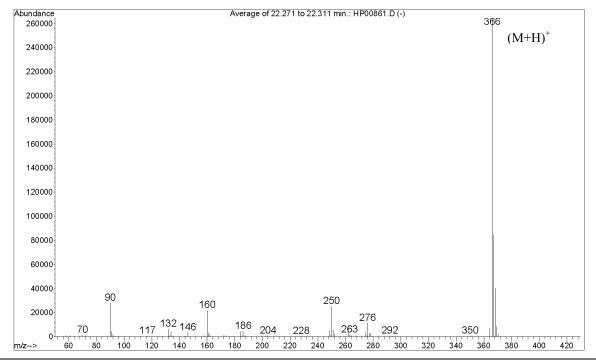
Abundance 900000



C:\DATA\16\HP00861.D

Acquired : 24 Nov 2004 18:09 Sample Name: 1uL of CW-CK-1-127-6 using AcqMethod CW-CI-TM

(compound J) Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH3



CI mass spectrum of:

Compound 5 in Liquid sample L/31, aliquot CW-1-131-5-L Top:

Bottom: TMS derivative of the authentic reference standard of **Triethanolamine** trimethylsiloxyethyl)amine] corresponding to compound 5 (MW: 365)

[tris(2-

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 Sample code(s): L/31, LB/31 Compound number: 6								
Aliquot codes:								
Sample: CW-1-131-4-L		,	Blank:	CW-1-131	1-3-LB			
CC EI MC Mathad name	TMC	<u> </u>						
GC-EI-MS Method name:	TMS_A	4						
METHOD DESCRIPTION								
Instrument Manufacturer	Agilent 6890/5973 GC/MSD							
and Type:								
Carrier gas:	\square He \square N ₂ \square H ₂ \square Other:							
Flow rate:	☐ ml/min ⊠ 38 cm/s							
Flow control:	☐ Constant Pressure ☐ Constant Flow							
Injection mode:	\square Split \rightarrow Split ratio =							
	\boxtimes Splitless \rightarrow Splitless time = 0. 70 min.							
Injector temperature:	250 °C							
Column phase:	5% diphenyl 95% dimethyl polysiloxane							
Column Length x ID x	30 m x 0.25 mm x 0.25 μm							
Film thickness:								
GC temperature	70 °C (8 min), 8 °C/min, 300 °C (3 min)							
programme:								
Solvent delay time:	8 min		Scan ran		40-600 m/z			
Electron energy:	70 eV		Scan tim		0.7 s			
Ionisation polarity:								
	∐ Neg	gative						
Comments:								
ANALYSIS								
Compound identified as:		☐ Original compound						
		☐ Methyl ester derivative						
		☐ TBDMS (t-Butyldimethylsilyl) derivative						
		☐ TMS (Trimethylsilyl) derivative						
	☐ Other derivative:							
Retention parameter used for		\boxtimes Retention time (Rt)						
(peak) identification:		☐ Scan number						
⊠ Compared to reference chemical:		Source : Own Synthesis Commercial						
☐ Compared to library spectrum:		Source: OCAD (code:) NIST						
		☐ Wiley ☐ Own ☐ Other:						
☐ Not compared to reference		Intense ions in spectrum are explained; interpretation is						
chemical or library spectrum:		supported by the spectral information derived from						
		closely 1	related che	emical(s):				
Comments:								

EI chromatograms supporting identification of compound 6; TIC on left; EIC (m/z 114) on right.

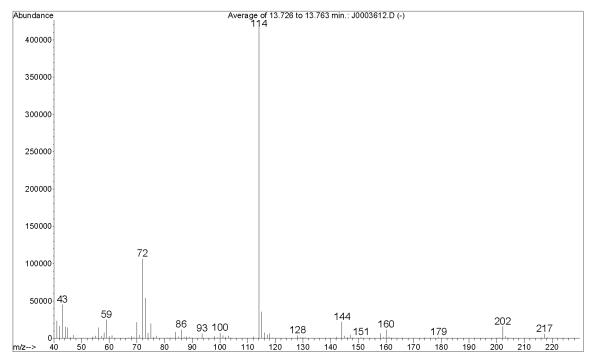
Top: Chromatograms of Liquid blank, aliquot **CW-1-131-3-LB** from **LB/31**.

Center: Chromatograms of Liquid sample, aliquot CW-1-131-4-L from L/31, retention time 13.74 min.

Bottom: Chromatograms of TMS derivative of the standard of 2-(N,N-Diisopropylamino)ethanol

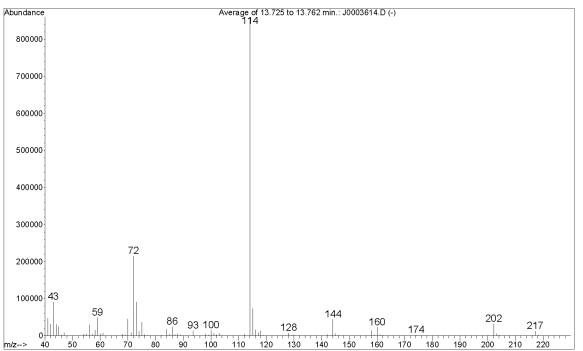
[2-(N,N-Diisopropylamino)ethyl trimethylsilyl ether], retention time 13.75 min.

File : C:\DATA\16\J0003612.D
Acquired : 29 Nov 2004 18:17
Sample Name: CW-1-131-4-L
Misc Info : using AcqMethod TMS_A



: C:\DATA\16\J0003614.D Acquired : 29 Nov 2004 19:47 Sample Name: CW-CK-1-128-2 using AcqMethod TMS_A

Misc Info



EI mass spectrum of:

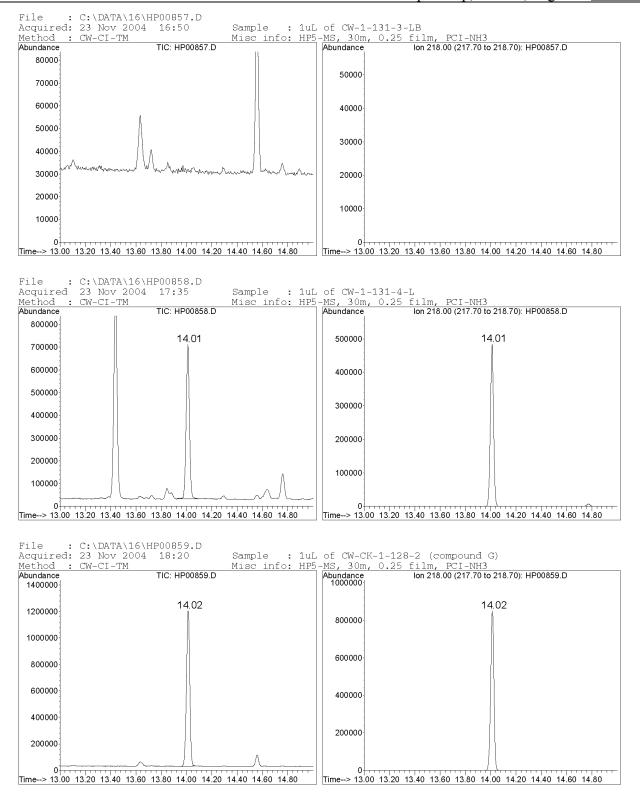
Compound 6 in Liquid sample L/31, aliquot CW-1-131-4-L Top:

Bottom: TMS derivative of the authentic reference standard of 2-(N,N-Diisopropylamino)ethanol [2-(N,N-Diisopropylamino)ethyl trimethylsilyl ether] corresponding to compound 6

(MW: 217)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 Sample code(s): L/31, LB/31 Compound number: 6								
Aliquot codes:								
Sample: CW-1-131-4-L		-	Blank:	CW-1-131	1-3-LB			
GC-CI-MS Method name:	CW-C	I-TM						
METHOD DESCRIPTION								
Instrument Manufacturer	Agilent 6890/5973 GC/MSD							
and Type:	Agnetit 0070/37/3 OC/MSD							
Carrier gas:	\square He \square N ₂ \square H ₂ \square Other:							
Flow rate:	\square ml/min \boxtimes 32 cm/s							
Flow control:	☐ Constant Pressure ☐ Constant Flow							
Injection mode:	\square Split \rightarrow Split ratio =							
· ·	\boxtimes Splitless \rightarrow Splitless time = 0.75 min.							
Injector temperature:	250 °C							
Column phase:	5% diphenyl 95% dimethyl polysiloxane							
Column Length x ID x	30 m x 0.25 mm x 0.25 μm							
Film thickness:								
GC temperature	70 °C (8 min), 8 °C/min, 300 °C (3 min)							
programme:								
Reaction gas:	☐ Methane ☐ Isobutane ☐ Ammonia ☐ Other:							
Solvent delay time:	8 min		Scan range:		50-550 m/z			
Electron energy:	235 eV		Scan time:		0.35 s			
Ionisation polarity:	⊠ Pos		Mass resolution:		0.7 u			
Comment	☐ Neg	gative						
Comments:								
ANALYSIS		<u> </u>						
Compound identified as:		☐ Original compound						
		☐ Methyl ester derivative						
		☐ TBDMS (t-Butyldimethylsilyl) derivative						
	☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:							
D-44:								
Retention parameter used for (peak) identification:								
☐ Compared to reference chemical:		Source : Own Synthesis Commercial						
Compared to library spectrum:		Source: OCAD (code:) NIST						
compared to notary spectrum.		Wiley □ Own □ Other:						
Not compared to reference		Intense ions in spectrum are explained; interpretation is						
chemical or library spectrum:		supported by the spectral information derived from						
			related che					
Comments:								



CI chromatograms supporting identification of compound 6; TIC on left; EIC (m/z 218) on right.

Top: Chromatograms of Liquid blank, aliquot **CW-1-131-3-LB** from **LB/31**.

Center: Chromatograms of Liquid sample, aliquot CW-1-131-4-L from L/31, retention time 14.01 min.

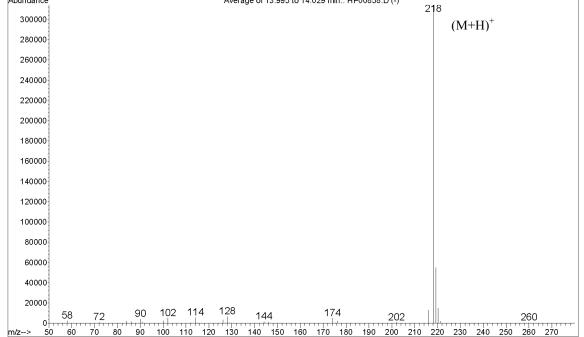
Bottom: Chromatograms of TMS derivative of the standard of **2-(N,N-Diisopropylamino)ethanol** [2-

(N,N-Diisopropylamino)ethyl trimethylsilyl ether], retention time **14.02** min.

File : C:\DATA\16\HP00858.D
Acquired : 23 Nov 2004 17:35 using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-1-131-4-L
Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH3

Abundance Average of 13.995 to 14.029 min.: HP00858.D(-)

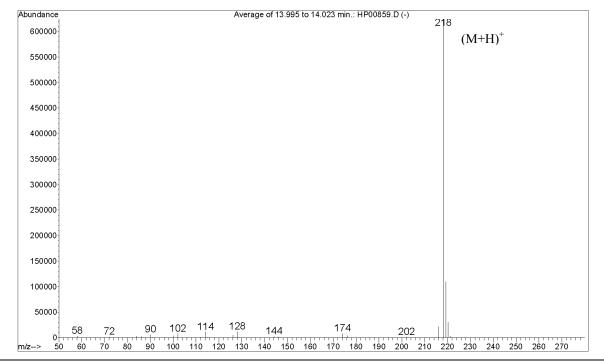
218
300000



File : C:\DATA\16\HP00859.D

Acquired : 23 Nov 2004 18:20 using AcqMethod CW-CI-TM

Sample Name: 1uL of CW-CK-1-128-2 (compound G) Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH3



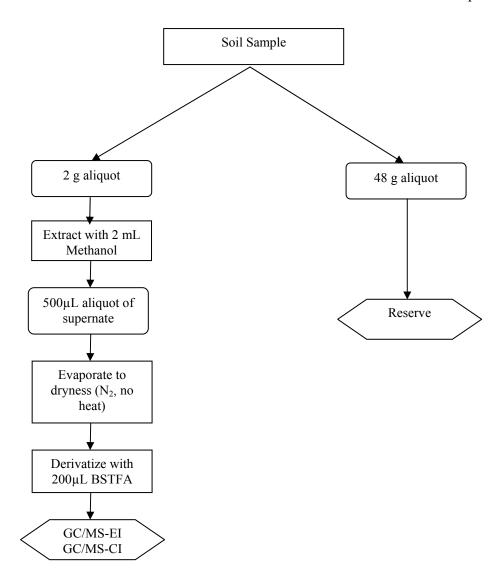
CI mass spectrum of:

Top: Compound 6 in Liquid sample L/31, aliquot CW-1-131-4-L

Bottom: TMS derivative of the authentic reference standard of 2-(N,N-Diisopropylamino)ethanol [2-

(N,N-Diisopropylamino)ethyl trimethylsilyl ether corresponding to compound 6 (MW: 217)

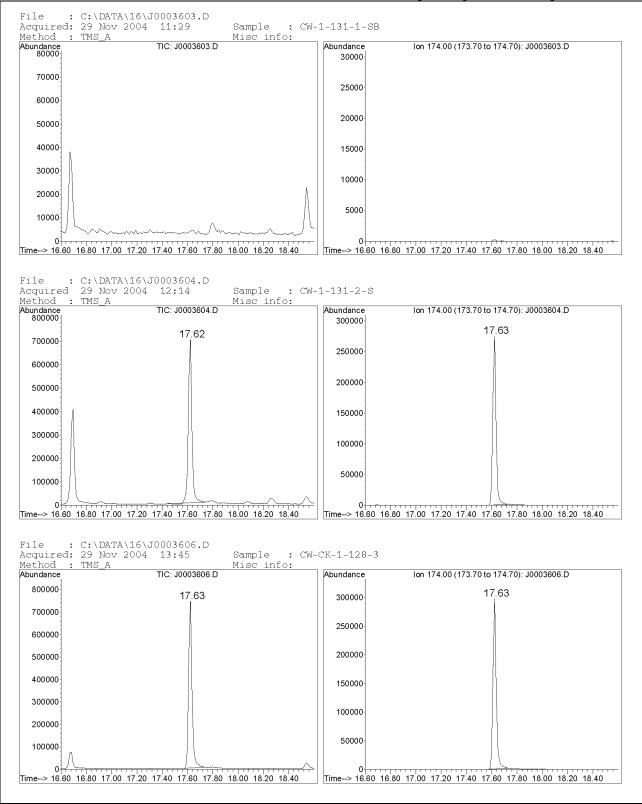
Laboratory code: 31 Sample code(s): S/31 Sample Blank code: SB/31										
. Sample pr	1						,			
Sample/ Aliquot Code	Specification of Sample/ Type of Sample Preparation	Amount/ Volume	Sample Pr	eparation Procedures		End Volume	Resulting Aliquot Code	Analytical Technique(s)		
Aliquot Code S/31 SB/31	TMS derivative of methanol extract of sample	2 g	for 15 min 500µL of	hanol was added to 2 g of soil an autes followed by 4 minutes of ce the supernate was dried (N2, no las added and heated at 60°C for 2	200 μL	CW-1-131-2-S CW-1-131-1-SB	GC/MS-EI GC/MS-CI			



Note: This flowchart is for visualization only; see the preceding sample preparation description page for sample aliquot numbers

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ample co	ode(s): <u>S/</u>	31, SB/31	_ Comp	ound number:	<u>7</u>
Aliquot codes:						
Sample: CW-1-131-2-S		,	Blank:	CW-1-131	1-1-SB	
GC-EI-MS Method name:	TMS A					
GC-EI-MS Method hame.	1 W15_E	1				
METHOD DESCRIPTION						
Instrument Manufacturer	Agilent	6890/5973	GC/MSD			
and Type:						
Carrier gas:	⊠ He	\square N ₂	H_2	Other:		
Flow rate:		ml/min	\boxtimes 38			
Flow control:		stant Pressu		Constant F	low	
Injection mode:			Split ratio			
		tless →	Splitless ti	ime = 0.70	min.	
Injector temperature:	250 °C					
Column phase:	-	henyl 95% d		olysiloxane		
Column Length x ID x	30 m x	0.25 mm x 0).25 μm			
Film thickness:						
GC temperature	70 °C (8 min), 8 °C	/min, 300	°C (3 min)		
programme:	0 .				1.0.500	
Solvent delay time:	8 min		Scan range:		40-600 m/z	
Electron energy:	70 eV	•.•	Scan time:		0.7 s	
Ionisation polarity:	⊠ Pos		Mass resolution:		0.7 u	
Comments	☐ Neg	gative				
Comments:						
ANALYSIS						
Compound identified as:		☐ Original compound				
		☐ Methyl ester derivative				
		☐ TBDMS (t-Butyldimethylsilyl) derivative				
		☐ TMS (Trimethylsilyl) derivative				
		☐ Other derivative:				
Retention parameter used for	or	☐ Retention time (Rt)				
(peak) identification:	☐ Scan nu	mber				
□ Compared to reference che	mical:	Source:	Own Sy	nthesis		
☐ Compared to library spectr	um:	Source:	☐ OCAL	O (code:) NIST	
			☐ Wiley			
☐ Not compared to reference					lained; interpretati	
chemical or library spectru	m:				rmation derived fi	rom
		closely 1	related che	emical(s):		
Comments:						



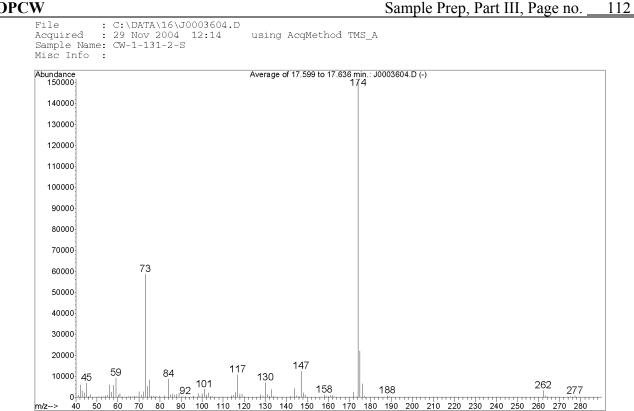
EI chromatograms supporting identification of compound 7; TIC on left; EIC (m/z 174) on right.

Top: Chromatograms of Soil blank, aliquot CW-1-131-1-SB from SB/31.

Center: Chromatograms of Soil sample, aliquot CW-1-131-2-S from S/31, retention time 17.62 min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Ethyldiethanolamine**

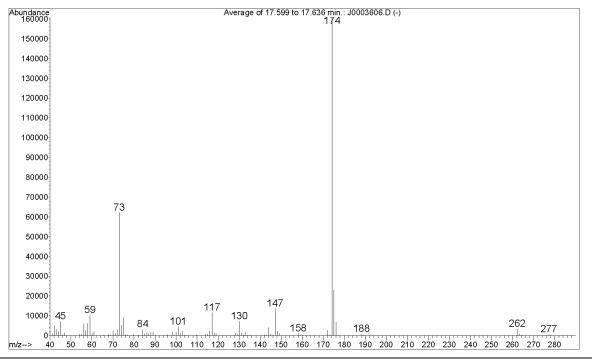
[Bis(2-trimethylsilyloxyethyl)ethylamine], retention time 17.63 min.



C:\DATA\16\J0003606.D

File : C:\DATA\16\J0 Acquired : 29 Nov 2004 : Sample Name: CW-CK-1-128-3 using AcqMethod TMS_A

Misc Info



EI mass spectrum of:

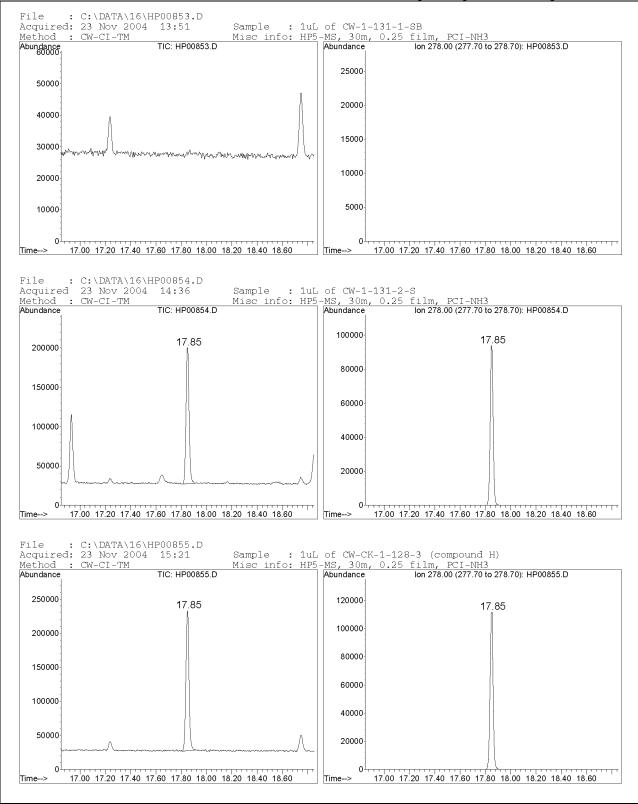
Compound 7 in Soil sample S/31, aliquot CW-1-131-2-S Top:

Bottom: TMS derivative of the authentic reference standard of Ethyldiethanolamine

[Bis(2-trimethylsilyloxyethyl)ethylamine] corresponding to compound 7 (MW: 277)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: <u>51</u> S	ample code(s): <u>s</u>	5/31, SD/31	Comp	ound number: <u>/</u>				
Aliquot codes:								
Sample: CW-1-131-2-S		Blank:	CW-1-131	-1-SB				
GC-CI-MS Method name:	CW-CI-TM							
METHOD DESCRIPTION								
Instrument Manufacturer	Agilent 6890/5973	3 GC/MSD						
and Type:								
Carrier gas:		\boxtimes He \square N ₂ \square H ₂ \square Other:						
Flow rate:	ml/min		2 cm/s					
Flow control:	☐ Constant Press	ure 🖂	Constant F	low				
Injection mode:	\square Split \rightarrow	Split ratio	=					
	\boxtimes Splitless \rightarrow	Splitless t	ime = 0.75 r	nin.				
Injector temperature:	250 °C							
Column phase:	5% diphenyl 95%	dimethyl p	olysiloxane					
Column Length x ID x	30 m x 0.25 mm x	0.25 μm						
Film thickness:		•						
GC temperature	70 °C (8 min), 8 °C	C/min. 300	°C (3 min)					
programme:	(6)							
Reaction gas:	☐ Methane ☐	Isobutane	X Ammo	nia				
Solvent delay time:	8 min	Scan rai		50-550 m/z				
Electron energy:	235 eV	Scan tin		0.35 s				
Ionisation polarity:			solution:	0.7 u				
Tomsulon pourry.	□ Negative							
Comments:				I				
ANALYSIS	i							
Compound identified as:	_	☐ Original compound						
	— ·	☐ Methyl ester derivative						
		☐ TBDMS (t-Butyldimethylsilyl) derivative						
	`	☐ TMS (Trimethylsilyl) derivative						
		☐ Other derivative:						
Retention parameter used for		⊠ Retention time (Rt)						
(peak) identification:	☐ Scan n	umber						
⊠ Compared to reference che	mical: Source:	Own Sy	nthesis					
☐ Compared to library spectr		☐ OCAI	O (code:) NIST				
		☐ Wiley	Ow	n Other:				
Not compared to reference	Intense io	ns in spectr	um are expl	ained; interpretation is				
chemical or library spectru		ted by the s	pectral infor	rmation derived from				
		related che						
Comments:								



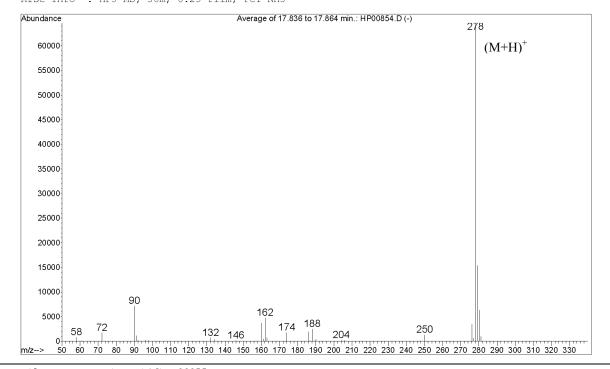
CI chromatograms supporting identification of compound 7; TIC on left; EIC (m/z 278) on right.

Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

Center: Chromatograms of Soil sample, aliquot CW-1-131-2-S from S/31, retention time 17.85 min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Ethyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)ethylamine], retention time **17.85** min.

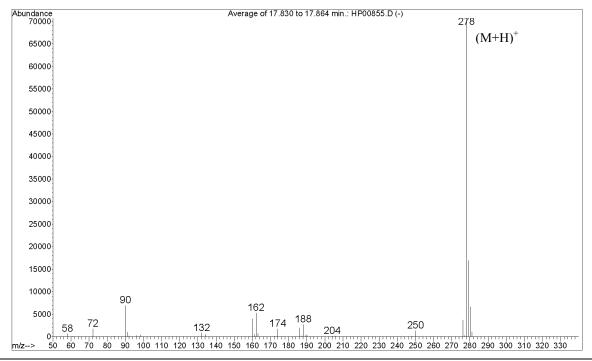
File : C:\DATA\16\HP00854.D
Acquired : 23 Nov 2004 14:36 using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-1-131-2-S
Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH3



File : C:\DATA\16\HP00855.D

Acquired : 23 Nov 2004 15:21 using AcqMethod CW-CI-TM

Sample Name: 1uL of CW-CK-1-128-3 (compound H) Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH3



CI mass spectrum of:

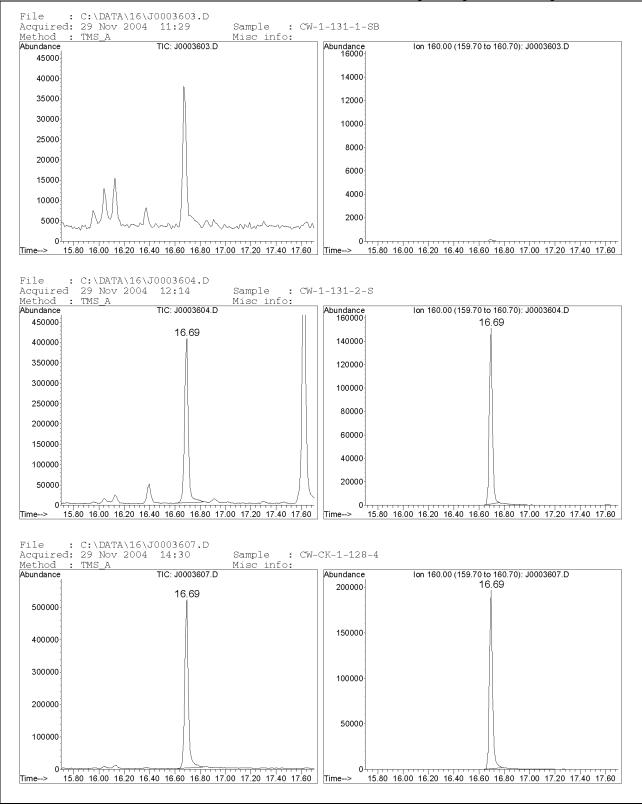
Top: Compound 7 in Soil sample S/31, aliquot CW-1-131-2-S

Bottom: TMS derivative of the authentic reference standard of **Ethyldiethanolamine**

[Bis(2-trimethylsilyloxyethyl)ethylamine]corresponding to compound 7 (MW: 277)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ampie co	oae(s): <u>8/</u>	31, SB/31	Comp	ound number: <u>8</u>	
Aliquot codes:						
Sample: CW-1-131-2-S			Blank:	CW-1-131	l-1-SB	
CC EI MC M-4L - J	TMC	<u> </u>				
GC-EI-MS Method name:	TMS_A	4				
METHOD DESCRIPTION						
Instrument Manufacturer	Agilent	6890/5973	GC/MSD			
and Type:						
Carrier gas:	⊠ He	\square N ₂	\square H ₂	Other:		
Flow rate:		ml/min	⊠ 38	3 cm/s		
Flow control:	☐ Con	stant Pressu	re 🖂	Constant F	low	
Injection mode:	☐ Spli	$t \rightarrow$	Split ratio	=		
	⊠ Spli	tless →	Splitless t	ime = 0.70	min.	
Injector temperature:	250 °C					
Column phase:	5% dip	henyl 95% d	limethyl p	olysiloxane		
Column Length x ID x	30 m x	0.25 mm x 0).25 μm			
Film thickness:						
GC temperature	70 °C (8 min), 8 °C	min, 300	°C (3 min)		
programme:						
Solvent delay time:	8 min		Scan range:		40-600 m/z	
Electron energy:	70 eV		Scan time: 0.		0.7 s	
Ionisation polarity:	⊠ Pos		Mass resolution:		0.7 u	
	☐ Neg	gative				
Comments:						
ANALYSIS						
Compound identified as:		☐ Original compound				
		☐ Methyl ester derivative				
		TBDMS (t-Butyldimethylsilyl) derivative				
		,	-	lyl) derivati	ve	
		☐ Other derivative:				
Retention parameter used for	or	Retention time (Rt)				
(peak) identification:		☐ Scan nu	ımber			
	mical:	Source:		nthesis		
☐ Compared to library spectr	um:	Source:		O (code:) NIST	
			☐ Wiley			
☐ Not compared to reference			-	-	ained; interpretation is	
chemical or library spectru	m:		-	-	rmation derived from	
		closely	related che	emical(s):		
Comments:						



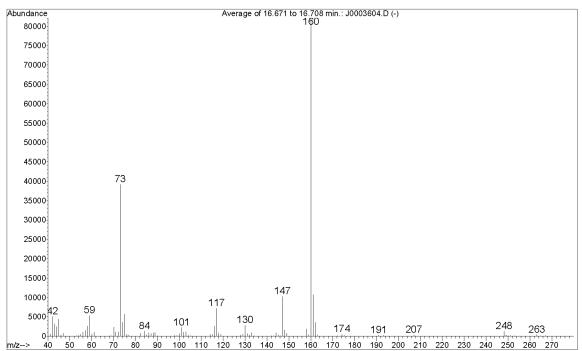
EI chromatograms supporting identification of compound 8; TIC on left; EIC (m/z 160) on right.

Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

Center: Chromatograms of Soil sample, aliquot CW-1-131-2-S from S/31, retention time 16.69 min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Methyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)methylamine], retention time **16.69** min.

File : C:\DATA\16\J0003604.D
Acquired : 29 Nov 2004 12:14
Sample Name: CW-1-131-2-S
Misc Info : using AcqMethod TMS_A

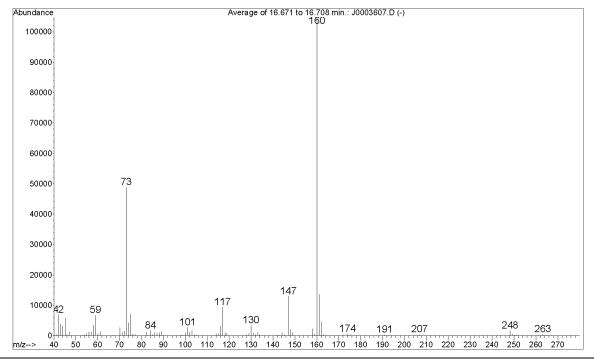


C:\DATA\16\J0003607.D

Acquired 29 Nov 2004 using AcqMethod TMS_A

Sample Name: CW-CK-1-128-4

Misc Info



EI mass spectrum of:

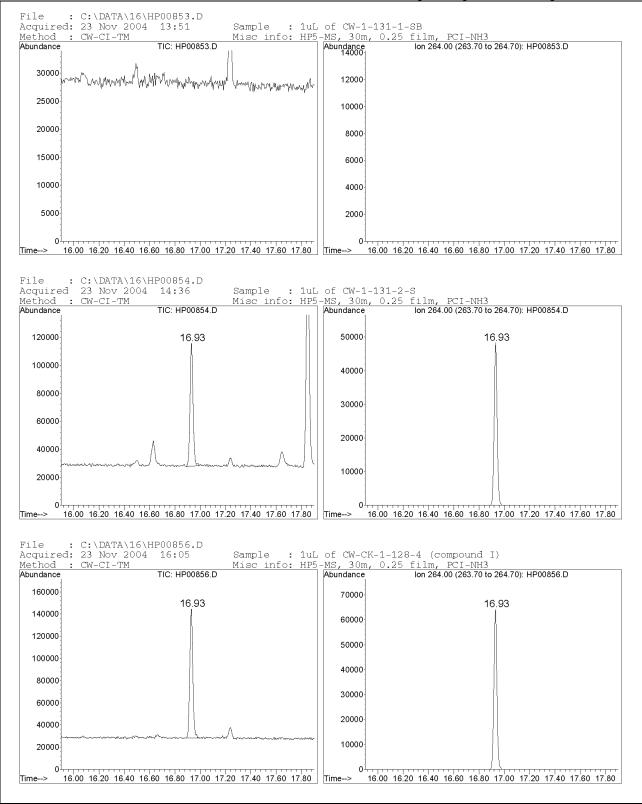
Compound 8 in Soil sample S/31, aliquot CW-1-131-2-S Top:

Bottom: TMS derivative of the authentic reference standard of Methyldiethanolamine

[Bis(2-trimethylsilyloxyethyl)methylamine] corresponding to compound 8 (MW: 263)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: <u>51</u> S	ampie code(s):	<u> 5/31, 5D/31</u>	Comp	ound number: <u>8</u>		
Aliquot codes:						
Sample: CW-1-131-2-S		Blank:	CW-1-131	1-1-SB		
GC-CI-MS Method name:	CW-CI-TM					
	O VV OI 1111					
METHOD DESCRIPTION	4 1 4 6000/505	12 CC/14CD				
Instrument Manufacturer	Agilent 6890/597	3 GC/MSD				
and Type:						
Carrier gas:	\square He \square N ₂	\square H ₂	Other:			
Flow rate:	ml/mir		2 cm/s	1		
Flow control:	Constant Pres		Constant F	low		
Injection mode:	\square Split \rightarrow	1				
	\boxtimes Splitless \rightarrow	Splitless t	ime = 0.75 r	nin.		
Injector temperature:	250 °C					
Column phase:	5% diphenyl 95%	dimethyl p	olysiloxane			
Column Length x ID x	30 m x 0.25 mm >	x 0.25 μm				
Film thickness:						
GC temperature	70 °C (8 min), 8 °	°C/min, 300	°C (3 min)			
programme:						
Reaction gas:	☐ Methane ☐	Isobutane	⊠ Ammo	nia 🗌 Other:		
Solvent delay time:	8 min	Scan rai	nge:	50-550 m/z		
Electron energy:	235 eV	Scan tin	1e:	0.35 s		
Ionisation polarity:	□ Positive	Mass re	solution:	0.7 u		
	☐ Negative					
Comments:		•				
ANALYSIS	100					
Compound identified as:		☐ Original compound				
		Methyl ester derivative				
		☐ TBDMS (t-Butyldimethylsilyl) derivative				
		☐ TMS (Trimethylsilyl) derivative				
		Other derivative:				
Retention parameter used for		Retention time (Rt)				
(peak) identification:		number				
□ Compared to reference che	mical: Source:	Own S	ynthesis			
☐ Compared to library spectr	um: Source:		D (code:) 🗌 NIST		
		☐ Wiley				
Not compared to reference				ained; interpretation is		
chemical or library spectru				rmation derived from		
	closel	y related che	emical(s):			
Comments:						



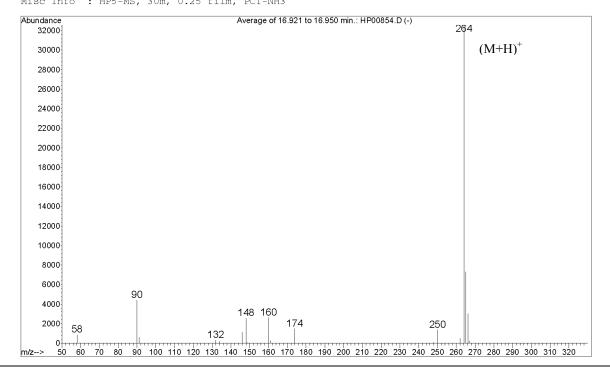
CI chromatograms supporting identification of compound 8; TIC on left; EIC (m/z 264) on right.

Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

Center: Chromatograms of Soil sample, aliquot CW-1-131-2-S from S/31, retention time 16.93 min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Methyldiethanolamine** [Bis(2-trimethylsilyloxyethyl)methylamine], retention time **16.93** min.

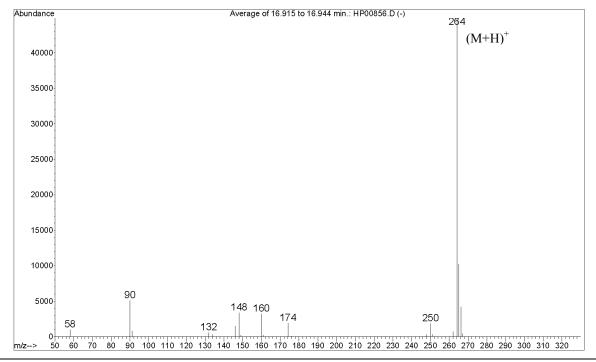
File : C:\DATA\16\HP00854.D
Acquired : 23 Nov 2004 14:36 using AcqMethod CW-CI-TM
Sample Name: 1uL of CW-1-131-2-S
Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH3



File : C:\DATA\16\HP00856.D

Acquired : 23 Nov 2004 16:05 using AcqMethod CW-CI-TM

Sample Name: 1uL of CW-CK-1-128-4 (compound I) Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH3



CI mass spectrum of:

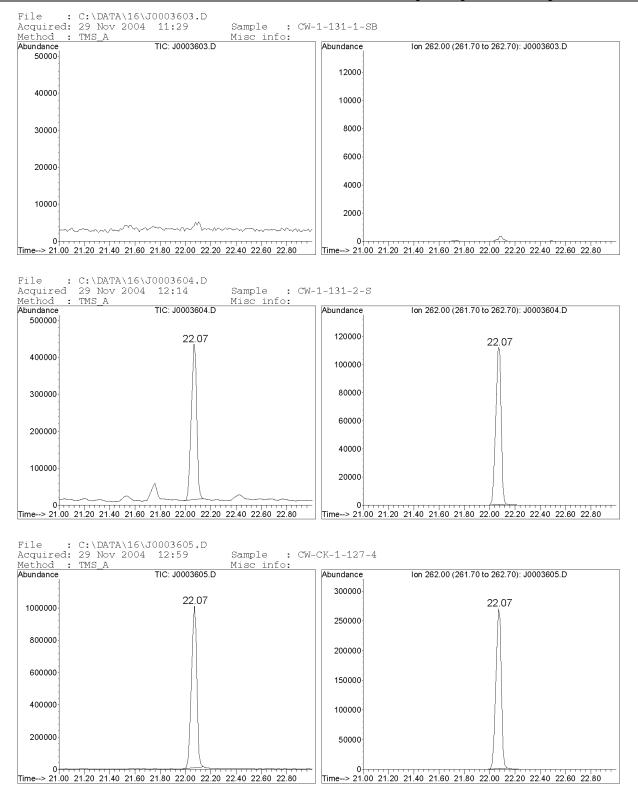
Top: Compound 8 in Soil sample S/31, aliquot CW-1-131-2-S

Bottom: TMS derivative of the authentic reference standard of **Methyldiethanolamine**

[Bis(2-trimethylsilyloxyethyl)methylamine] corresponding to compound 8 (MW: 263)

GC-EI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ample co	ode(s): <u>S/</u>	31, SB/31	_ Comp	ound number:	9
Aliquot codes:						
Sample: CW-1-131-2-S		,	Blank:	CW-1-131	1-1-SB	
CC EI MC Mathad name	TMC	<u> </u>				
GC-EI-MS Method name:	TMS_A	4				
METHOD DESCRIPTION						
Instrument Manufacturer	Agilent	6890/5973	GC/MSD			
and Type:						
Carrier gas:	⊠ He	\square N_2	\square H ₂	Other:		
Flow rate:		ml/min	⊠ 38			
Flow control:	☐ Con	stant Pressu	re 🖂	Constant F	low	
Injection mode:	☐ Spli	$t \rightarrow$	Split ratio	=		
	⊠ Spli	tless →	Splitless ti	ime = 0.70 r	nin.	
Injector temperature:	250 °C					
Column phase:	5% dip	henyl 95% d	limethyl po	olysiloxane		
Column Length x ID x	30 m x	0.25 mm x 0).25 μm			
Film thickness:						
GC temperature	70 °C (8 min), 8 °C	/min, 300	°C (3 min)		
programme:			1		1	
Solvent delay time:	8 min		Scan range:		40-600 m/z	
Electron energy:	70 eV				0.7 s	
Ionisation polarity:	⊠ Pos		Mass resolution:		0.7 u	
	∐ Neg	gative				
Comments:						
ANALYSIS						
Compound identified as:		☐ Original compound				
		☐ Methyl ester derivative				
		☐ TBDMS (t-Butyldimethylsilyl) derivative				
		☐ TMS (Trimethylsilyl) derivative				
		☐ Other derivative:				
Retention parameter used for	or	⊠ Retention time (Rt)				
(peak) identification:		☐ Scan nu	mber			
□ Compared to reference che	mical:	Source:	Own Sy	nthesis		
☐ Compared to library spectr	um:	Source:		O (code:) NIST	
			☐ Wiley			
☐ Not compared to reference					ained; interpretati	
chemical or library spectru	m:				rmation derived fi	rom
		closely 1	related che	emical(s):		
Comments:						



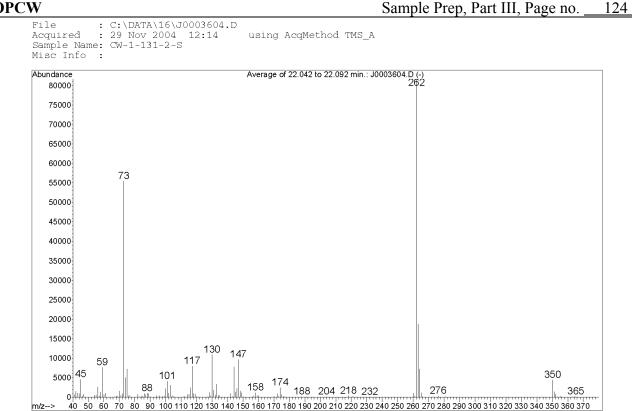
EI chromatograms supporting identification of compound 9; TIC on left; EIC (m/z 262) on right.

Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

Center: Chromatograms of Soil sample, aliquot CW-1-131-2-S from S/31, retention time 22.07 min.

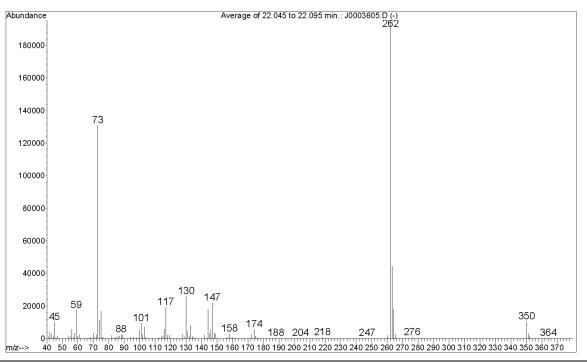
Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Triethanolamine**[trie(2, trimathylailogy/athyl)eminal retention time 22,07 min

[tris(2-trimethylsiloxyethyl)amine], retention time **22.07** min.



File : C:\DATA\16\J0003605.D Acquired : 29 Nov 2004 12:59 Sample Name: CW-CK-1-127-4 using AcqMethod TMS_A

Misc Info



EI mass spectrum of:

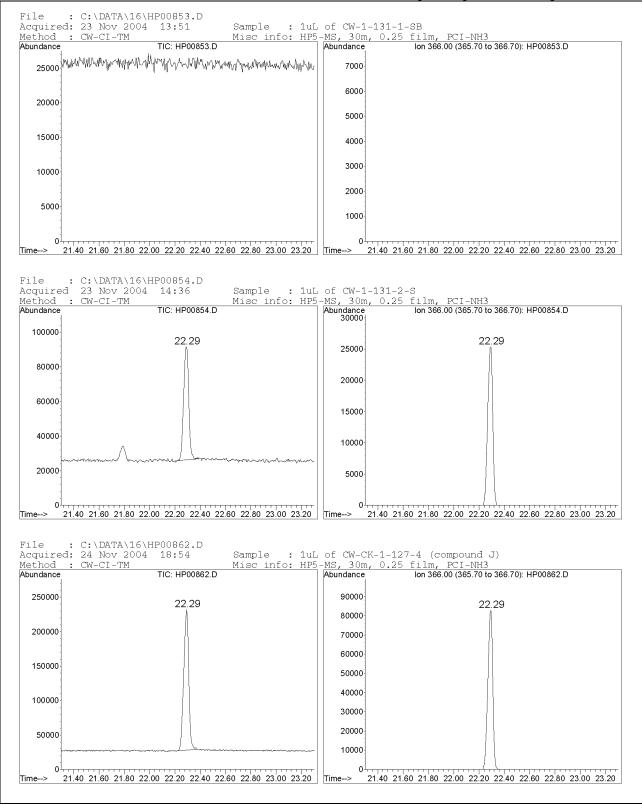
Compound 9 in Soil sample S/31, aliquot CW-1-131-2-S Top:

Bottom: TMS derivative of the authentic reference standard of Triethanolamine

[tris(2-trimethylsiloxyethyl)amine] corresponding to compound 9 (MW: 365)

GC-CI-MS TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 31 S	ample code	(s): <u>S</u>	/31, SB/31	_ Comp	ound number:	9
Aliquot codes:						
Sample: CW-1-131-2-S			Blank:	CW-1-131	1-1-SB	
GC-CI-MS Method name:	CW-CI-T	М				
METHOD DESCRIPTION						
Instrument Manufacturer	Agilent 689	90/5973	GC/MSD			
and Type:						
Carrier gas:	⊠ He	\square N_2	\square H ₂	Other:		
Flow rate:		ml/min	⊠ 32			
Flow control:	☐ Constan	nt Pressu	ure 🖂	Constant F	low	
Injection mode:	☐ Split	\rightarrow	Split ratio	=		
	Splitles	$s \rightarrow$	Splitless ti	me = 0.75 r	nin.	
Injector temperature:	250 °C					
Column phase:	5% diphen	yl 95% (dimethyl po	olysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 μm					
GC temperature	70 °C (8 m	in), 8 °C	C/min, 300	°C (3 min)		
programme:						
Reaction gas:	☐ Methan	e 🗌 I	sobutane	⊠ Ammo	nia 🗌 Other:	
Solvent delay time:	8 min		Scan ran	ige:	50-550 m/z	
Electron energy:	235 eV		Scan time:		0.35 s	
Ionisation polarity:	□ Positive	e	Mass resolution		0.7 u	
	☐ Negativ	/e				
Comments:						
ANALYSIS						
Compound identified as:		 ☐ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative: 				
Retention parameter used for (peak) identification:	or 🖺	□ Retention time (Rt) □ Scan number				
□ Compared to reference che	mical: So	urce :	☐ Own Sy	nthesis		
Compared to library spectr		urce :) (code:) NIST on Other:	
Not compared to reference chemical or library spectru		Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):				
Comments:		·				



CI chromatograms supporting identification of compound 9; TIC on left; EIC (m/z 366) on right.

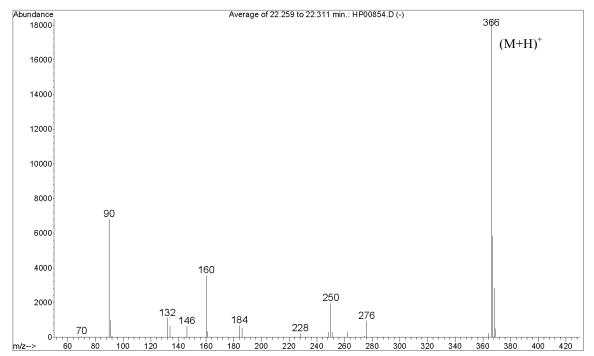
Top: Chromatograms of Soil blank, aliquot **CW-1-131-1-SB** from **SB/31**.

Center: Chromatograms of Soil sample, aliquot CW-1-131-2-S from S/31, retention time 22.29 min.

Bottom: Chromatograms of TMS derivative of the authentic reference standard of **Triethanolamine**[trie(2 trimethylailogyathylamina] retention time 22 20 min

[tris(2-trimethylsiloxyethyl)amine], retention time **22.29** min.

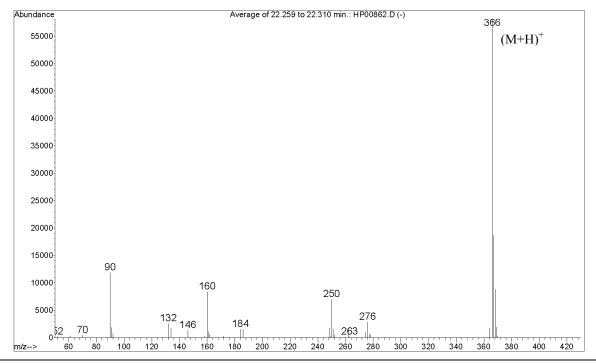
File : C:\DATA\16\HP00854.D
Acquired : 23 Nov 2004 14:36 using Acc
Sample Name: 1uL of CW-1-131-2-S
Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH3 using AcqMethod CW-CI-TM



C:\DATA\16\HP00862.D

24 Nov 2004 using AcqMethod CW-CI-TM Acquired

Sample Name: 1uL of CW-CK-1-127-4 (compound J) Misc Info : HP5-MS, 30m, 0.25 film, PCI-NH3



CI mass spectrum of:

Compound 9 in Soil sample S/31, aliquot CW-1-131-2-S Top:

Bottom: TMS derivative of the authentic reference standard of **Triethanolamine** trimethylsiloxyethyl)amine] corresponding to compound 9 (MW: 365)

[tris(2-